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DETERMINATION OF THE SPECIFIC SURFACE AREA OF PARTICLES BY MEANS OF HINDERED SETTLING

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BY MEANS OF HINDERED SETTLING

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PREFACE

I wish to take this opportunity to acknowledge the invaluable contributions of Prof. J. M. DallaValle, who conceived and directed this research. His patience and encouragement were a constant source of inspiration throughout this investigation.

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SUMMARY

DETERMINATION OF THE SPECIFIC SURFACE OF PARTICLES BY MEANS OF HINDERED SETTLING

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The hindered settling of suspended particles may be regarded as a characteristic of concentrated suspensions. It is the type of settling frequently encountered in chemical engineering operations.

Hindered or "line" settling is that form of settling exhibited by concentrated suspensions when the suspended material settles en masse. The distribution of variously sized particles tends to remain the same while the density of the suspension increases. This causes the clear excess suspending medium to be separated from the suspension, as time increases, by a distinct interface or "line". Given enough time, the suspension will settle to an ultimate volume that will remain at a constant value. This mode of settling is distinctly different from "free" or Stokes settling wherein the individual particles of a suspension will fall at a rate proportional to their diameters in accordance with Stokes' or Newton's law of settling.

There have been numerous attempts to explain the nature of hindered

settling, but because of the complexity of the variables involved, no completely satisfactory theory has thus far been suggested. Most treatments of the problem have been modifications of Stokes' law which is valid only for a single sphere in an infinite expanse of a viscous fluid. None of these modifications have sufficient generality to cope completely with the problem.

A recent paper by Allison and Murray (1951) and the experiences obtained with the production of powdered material, have indicated that the hindered settling rates of suspensions are ultimately connected with the specific surface area of the suspended material. The Allison and Murray paper proposed a method for obtaining the specific surface of a powder from the behavior under hindered settling. The basic equation employed was the well-known Kozeny equation for the flow of a fluid through a packed bed. It was felt that the limitations imposed on this equation represented a serious drawback to the general applicability of the method.

This state of affairs prompted the present investigation, which is based on an empirical approach to the hindered settling behavior of disperse methyl alcohol suspensions of powdered materials. In all, 17 materials with specific gravities ranging from 2.71 to 11.14 were tested. The concentration of the suspensions employed varied from 300 to 3338 g./l. Methyl alcohol was employed as the suspending medium since it gave disperse suspensions of most materials without the addition of surface active agents. Water would not do this. The use of these surface active ingredients was not desirable, since their effect on hindered settling is not well known. They are, however, known to increase the

ultimate settled volume of a suspension. Disperse suspensions were required because in such cases, degree of flocculation is not an added variable (which is difficult to characterize).

Graduated mixing cylinders of 100 ml. capacity were used as the settling chambers. Powdered material was weighed and put into the cylinder. Methyl alcohol was then added to form a suspension whose concentration fell within the permissable range for hindered settling. Data of volume of suspension as a function of time were taken at room temperature.

Correlation of the observed data was based upon the dimensionless ratio, $V/(V_o - V)$, where V_o is the initial volume of the suspension and V is the suspension volume at any time, t. Only ratios of volume are significant for obtaining a general correlation that will be independent of the dimensions of the settling chamber.

The specific surface areas of the powders were obtained from the sedimentation data by the method of Allison and Murray. In addition, the gas permeametric method was employed for one material. These specific surface areas covered a range of from roughly 0.02 to 0.20 $m.^2/g.$

The final form of the correlation for the hindered settling of disperse methyl alcohol suspensions of irregularly shaped approximately spherical particles is

$$\frac{V}{V_o - V} = \frac{H}{H_o - H} = 2.88\, S^{5.07}\, C^{2.79}\, t^{-1.12}$$

where H_o is the initial height of the suspension and H is the height of

the suspension at any time, S is the specific surface area in $m.^2/g.$, C is the initial concentration of the suspension in g./l. and t is time in seconds.

It was found that this equation represented the experimental data within two to thirty per cent, depending upon the degree of reliability that one could attach to S. An error in S would produce a much larger error in the calculated value of V. Considering the fact that one usually does not have a value of S that is accurate to within 15 per cent (in reproducibility), the value of the above equation would appear to be very slight for the prediction of settling curves. It is the use of the equation for the estimation of the specific surface area of powdered materials that appears to be the most important application.

From limited experimental data on the hindered settling of a disperse methyl alcohol suspension of a powder at room temperature the simple and direct calculation of the specific surface area of the material is possible. It is believed that this method is at least as accurate and reliable (when used with proper caution) as the usual gas permeametric method and the newer Allison-Murray technique (which in many instances gives, at best, only an approximate value).

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CHAPTER I

INTRODUCTION

The hindered settling of suspended particles may be regarded as a characteristic of concentrated suspensions. It is a type of settling that is frequently encountered in chemical engineering operations. It is common to the thickening of slurries (as with Dorr thickeners) and the sedimenting of materials which have been collected by hydraulic methods.

Hindered settling may be defined as that form of settling achieved in concentrated suspensions when the suspended material settles uniformly; that is, when all particles tend to settle at the same rate. Hindered settling can be distinguished by the manner in which a mass of particles settle, leaving a clear fluid separated from the suspension by a sharp boundary line. This boundary has resulted in the term "line settling" also being applied to this type of settling. This mode of sedimentation is in contradistinction to the settling of dilute suspensions in which the larger particles tend to settle at a faster rate than do the smaller ones and there is exhibited in the body of the suspension a rather diffuse appearance as settling continues. In the latter case, settling takes place in accordance with Newton's or Stokes' law of settling.

There have been numerous attempts to explain the nature of

hindered settling but because of the complexity of the variables involved, no completely satisfactory theory has thus far been suggested. Most investigations of the problem, as will be shown in the next chapter, have been concerned with modifications of Stokes' law for the settling of a single particle in an infinite, viscous fluid. These modifications of Stokes' law have no validity in the present instance and in no case are they sufficiently general to explain the nature of hindered settling itself. There is, in fact, no simple approach to the problem at the present time.

Interest in investigating hindered settling stems from two considerations: (1) it has been found in many industries engaged in the production of powdered materials that hindered settling rates give a fairly practical means of determining average particle size; and (2) a recent publication by Allison and Murray (1951) gives reasonable proof that in many instances, the hindered settling rate of a given material is an index of its specific surface area. The latter has never been suspected of being an important variable in hindered settling studies. Because until now, no comprehensive study has been made of hindered settling using specific surface as a variable, a rather complete investigation of hindered settling from this point of view was judged to be of some importance. Should the hindered settling of a suspended material prove to be of value in estimating the specified surface area of the material, one would have at his disposal a simple and rapid technique which would prove to be of considerable value to those engaged in the preparation of powders (from the standpoint of control).

Since the method of Allison and Murray depends upon the appli-

cability of the Kozeny equation for the flow of a fluid through a packed bed, the method is limited by the restrictions imposed upon the Kozeny equation. It was therefore thought advisable to initiate a study utilizing an empirical approach, correlating the significant variables separately. The investigation which follows is based on this approach and as will be seen, demonstrates that the assumption of Allison and Murray as to the dependence of hindered settling upon the specific surface area of the material is warranted.

CHAPTER II

LITERATURE SURVEY

In a search for quantitative expressions relating to the fall of particles through a fluid one can go at least as far back as the work of Sir Isaac Newton. The fifth corollary of Proposition XXXV in his Principia (1686) states: "The resistance of a globe is in a ratio compounded of the duplicate ratio of the velocity and the duplicate ratio of the diameter and the ratio of the density of the medium." Expressing this idea in modern terminology,

$$F_D = C_D \, d \, A \, \frac{V^2}{2} \qquad (1)$$

where F_D is the drag force, d the density of the medium, A the projected area of the falling particle ($\pi D^2/4$ for a sphere), V the velocity of fall and C_D the coefficient of drag. Depending upon the values of C_D and d used, Equation 1 can be applied to bodies of any shape in any fluid. Unfortunately, Newton neglected to include the forces of internal friction in his formulation so that Equation 1, with a reasonable value of C_D, is valid only for Reynolds numbers, based upon the diameter of the particle, well beyond the viscous range.

Over one hundred years ago, Sir George Stokes (1850) published his famous treatise "On the Effect of the Internal Friction of Fluids

on the Motion of Pendulums." This paper is credited with being the initial disclosure of the mathematical statement for the nature of the drag force acting upon a sphere falling at uniform velocity through an infinite expanse of fluid. It is to be noted however, that the familiar result

$$F_D = 3\pi \mu V D \qquad (2)$$

(where μ is the viscosity of the fluid, D the diameter of the particle and V is the velocity) is obtained only as a special case of the more general pendulum problem. In fact, Stokes apparently thought this special case to be of little import, as there is no separate equation for it in his initial article. Indeed, it would have required a mathematician to deduce Equation 2 from his general pendulum formula. It is interesting to note that the term "viscosity" was not in scientific use at that time so that Stokes referred to this property of fluids as the "index of friction."

In a subsequent revision of his first paper, Stokes expanded the treatment to include this special case of the uniform motion of a sphere, in order to compare his theoretical equation with some experimental results obtained by Coulomb which served to confirm the general pendulum theory. The more familiar form of the Stokes equation for the terminal velocity of a particle falling under gravity,

$$V = \frac{g}{18\mu} (d_s - d_f) D^2 \qquad (3)$$

(where V is the terminal or "Stokes" velocity of fall, g the acceleration of gravity, d_s the density of the solid and d_f the density of the fluid with D and μ as before) was obtained by equating the force due to gravity ($\frac{4}{3}\pi r^3 g d_s$) to the buoyant force ($\frac{4}{3}\pi r^3 g d_f$) plus the drag force ($6\pi V\mu r$). It is to be noted that infinite expanse of fluid, smooth rigid spheres, no solvation or electro-viscous effect, no "slipping" of fluid at the surface of the sphere and low Reynolds number are assumed valid for Equations 2 or 3 to hold.

There have been numerous attempts to take into account violations of the above restrictions by applying suitable modifications to Stokes' law. Only those dealing with the effect of other particles upon the settling velocity will be discussed.

Cunningham (1910), from a hydrodynamical treatment of the specific case where the diameters of the settling particles are small compared to the mean free path of the fluid molecules, calculated that for particles 10^{-5} centimeters in diameter falling in air, Stokes' law gives a velocity that is approximately one half the actual value. Also, in considering a cloud of particles, he concluded that as long as the distance between particles was at least ten times the particle size, Stokes' law was not appreciably affected (all other restrictions being observed).

With what might be termed a practical approach to the problem of the settling of a suspension of particles in a fluid, Robinson

(1926) proposed modifications to the basic Stokes' equation that would allow it to be applied to concentrated suspensions of variously shaped particles. By his reasoning, the density of the fluid in Stokes' equation should be replaced by the density of the suspension since the buoyant force acting upon a particle would equal the weight of the suspension displaced. Likewise, the viscosity of the fluid is to be replaced by the viscosity of the suspension. Since various shapes of particles are to be covered by this modification, the constant 1/18 in Equation 3 (as derived for spherical particles) must be replaced by a constant, k, which should be unique for any given kind of solid, independent of the size of the particle, provided that the shapes of large and small particles are similar. Thus, the Robinson modification of Equation 3 takes on the form

$$V = \frac{k\, g\, (d_s - d_{sus.})\, D^2}{\mu_{sus.}} \qquad (4)$$

where the subscript "sus." refers to the designated property of the suspension. The diameter term, D, appearing above represents some average dimension of the particles involved.

If one considers the nature of the buoyant force acting upon a particle suspended in a fluid containing numerous other particles of the same size as the individual one under consideration, it will be seen that the weight of the fluid displaced is still the significant factor (as in Stokes' law). If the particle under consideration, however, is greatly larger than the other particle suspended in the fluid,

then the buoyant force acting upon the designated particle is equal to the weight of the suspension displaced. These two cases would represent the extremes encountered in dealing with a suspension of particles. If the material suspended were closely sized, the correct density to use in Equation 4 would be that of the fluid whereas, if the suspended material covered a range of sizes, the buoyant forces acting upon the individual particles would involve densities ranging from that of the fluid (for the smallest particles) up to that of the suspension (deleted of the largest particles). For some intermediate size of particle, the density to be used would be that of a suspension containing all of the particles smaller than that for which the buoyant force was desired. Thus the total buoyant force would be represented by a finite series, each term of which designated the force acting upon all particles of a particular size. Obviously, this series cannot be evaluated in practice. The use of the viscosity of the suspension is also open to criticism, as will be developed later. As mentioned by Eglof and McCabe (1937), Equation 4 applies only to highly concentrated suspensions.

In their paper, Eglof and McCabe recognize two main classifications of settling phenomena as applied to flocculated particles. The most common type consists of an initial constant rate period followed by a short transition period that leads into a final compression period. The second type is that having a constant settling rate followed by another constant rate period which leads to the ultimate height of the sediment with insignificant transition and compression periods. Their approach to the problem of the prediction of the

settling behavior of flocculated particles was to empirically correlate these initial constant rates with the concentration of the suspensions for the first type of settling only. The final result was that

$$R_o = \phi \left[\frac{r^2 (d_s - d_f)}{\mu} \frac{H_o}{H_u} (0.415 - V_u)^{1.4} \right] \quad (5)$$

where R_o is the initial rate of subsidence of the surface of the suspension of flocculated particles, H_o represents the initial height of the suspension, H_u represents the ultimate height of the suspension (at infinite time), V_u is the ultimate volume of the suspension and r the radius of the particles. ϕ designates some unknown function of the several variables (presented as a curve in the paper). The authors considered that $(0.415 - V_u)$ represented a measure of the intensity of the flocculation. Equation 5 was said to represent 80 per cent of the 76 runs within 20 per cent.

For the compression period of settling, Eglof and McCabe made use of a known logarithmic relationship

$$\log (H/H_u) = K_c \log B_t \quad (6)$$

where K_c and B are constants and t is time. K_c was substantially constant for any given material, regardless of the initial height of the suspension, the initial concentration or the temperature. In order to make use of these last two equations for the prediction of settling curves, it is practically necessary to have most of the data that would

enable one to draw the required curve without benefit of the equations. Also, the predicted curve was most often only within fair agreement with experimental results. One cause of this was probably the difficult task of obtaining a uniform degree of flocculation from one experiment to another.

In a series of three articles, Steinour (1944) has developed some rather interesting equations that apply to a variety of sedimentation conditions. The ultimate objective of the series was to develop an equation that would accurately represent such phenomena as the "bleeding" of cement pastes. In the first article, he applied hydromechanical considerations to the fall of a spherical particle through a suspension of like particles and obtained an expression quite similar to the familiar Kozeny equation for the flow of fluids through a packed bed of material. Steinour's equation for this is

$$Q = \frac{g}{18\mu} (d_s - d_f) D^2 \frac{e^3}{1-e} F(e) \qquad (7)$$

where Q is the velocity of the particles relative to a fixed horizontal plane, e is the porosity (or volume fraction of fluid) of the suspension and F(e) is a shape factor depending solely upon the porosity. Due to the nature of the boundary conditions placed upon Equation 7, F(e) must approach zero as e approaches infinity (the infinite dilution for Stokes' settling). However, at high concentrations, F(e) may remain practically constant. It is seen that Equation 7 can be expressed as

$$Q = V_s \frac{e^3}{1-e} F(e) \qquad (8)$$

where V_s is the Stokes velocity of fall of a particle through a fluid. To test this equation, Steinour conducted experiments upon closely sized spherical tapioca particles of diameter equal to 0.174 cm. suspended in an oil.

The results of these tests showed the equation to hold and F(e) to remain constant at about 0.123. The empirical equation that represented all of this data was

$$Q = V_s \, e^2 \, 10^{-1.82 \, (1-e)}. \qquad (9)$$

A comparison with Equation 8 shows the form of F(e). Microscopic glass spheres of about ten to twelve microns in diameter suspended in water were also tested with Equation 8. The equation applied to these particles, indicating that no new surface effects were taking place upon the reduction of particle diameter from that of the tapioca spheres to that of the glass spheres. Certain high concentration curves were disregarded in this generalization since they did not exhibit the uniform settling rate until well after the sedimentation had advanced. In the second article of the series, Steinour concerned himself with suspensions of uniform-sized angular particles. To test this case, emery particles having a maxium dimension of ten to twelve microns were suspended in water containing a wetting agent to preclude the possibility of flocculation. Here, Equation 8 did not apply. A correction term was necessary. This was designated as "w_i", referring to "immobile water" that was assumed to be dragged along with the settling particles. A similar correction to the Kozeny equation has been found necessary

for certain cases of the flow of water through clays. The expression governing suspensions of irregular, uniform-sized particles was given as

$$Q = \frac{0.123}{(1 - w_i)^2} V_s \frac{(e - w_i)^3}{1 - e} \qquad (10)$$

where the numerical value of F(e) and the correction for the "immobile water" has been applied to Equation 8. In the specific case of the emery mentioned above, w_i was found to equal 0.168. Since this value of w_i was found to hold for larger emery particles, Steinour concluded that the "immobile" liquid was actually stagnate liquid and not liquid bound to each particle as it settled.

In the third article of the series, Steinour turned his attention to flocculated suspensions of powders, each having a range of particle sizes. For this case, it became necessary to obtain a suitable Stokes velocity of fall for a suspension having a range of particular sizes. This was done by modifying the Stokes' equation to the expression

$$V_s = 2g \frac{(d_s - d_f)}{\mu S^2} \qquad (3a)$$

where S is the specific surface value of the powder, calculated as if each particle were a sphere having the same density as the original particle and the same rate of fall when alone in a large volume of the liquid. For uniform particles, S equals 3/r, where r is the radius

of the equal settling sphere. Numerically, S is obtained by experiment (the sedimentation analysis of a very dilute, non-flocculating suspension).

With this method of evaluating V_s, Equation 10 was tested utilizing data obtained for glass spheres (made by dropping glass powder through a flame), silica, limestone and shale in flocculated suspensions. The equation was validated, but flocculation resulted in an increase in w_i. It must be mentioned that the initial portions of the curves of subsidence of suspension level versus time did not appear as perfectly straight lines, but rather a noticeable curvature was exhibited in some cases. This was ignored and straight lines were drawn for this initial period.

Results at variance with Steinour's finding that w_i remained constant with varying particle size for disperse (i.e., non-flocculated) suspensions have appeared in the literature. Ward and Whitmore (1950) conducted a series of sedimentation studies upon methacrylate powders of four particle sizes ranging from 89 to 279 microns. They failed to obtain constant values of w_i. Additionally, a method of plotting the data as proposed by Steinour so as to obtain straight and parallel lines failed to do so in the case of the methacrylate powders. The data plotted as curved lines. Also, these authors found that w_i was substantially independent of the viscosity of the suspending medium. They agreed with Steinour's conclusion that the "immobile" liquid is not held to the particles by adsorption, but they found w_i to be dependent upon particle size. Since Steinour's emery was obtained from various sources, they concluded that perhaps the shape and surface

characteristics were different in each case.

In a recent article, Hawksley (1950) considered the settling rate of a concentrated suspension by assuming that an equilibrium particle arrangement would be established during settling. Previous hydrodynamical approaches to this subject had assumed a random distribution of the particles during settling. Hawksley's view was that while the distribution is undoubtedly random at the very beginning of settling, an orientation effect quickly sets in. This effect is due to the upward flowing liquid. It seems reasonable to assume that this upward flowing fluid will bring about a rearrangement of the successive layers so as to effect a minimum overall resistance or a maximum rate of settling. Thus, the particles will tend to become marshalled into vertical columns. When the particles are free to move in such a fashion, the rate of settling will increase steadily until an equilibrium arrangement has been produced. One would expect the transient state to persist for a greater length of time in a dilute suspension than in a concentrated suspension.

Assuming such an equilibrium arrangement of the particles, the general motion of these particles was examined in terms of the behavior of a single representative particle, obviating the need for a statistical treatment. Thus, the average rate of settling was obtained by considering the motion of a single particle in a homogeneous suspension having the same concentration at each point. This assumption is likely to be satisfied when the inter-particle distance is comparable with the particle diameter. It is to be noted that the mean distance between the surfaces of adjacent particles is only about twice their diameter

at concentration as low as five per cent by volume. At concentrations greater than ten per cent, the spacing is less than one diameter.

Hawksley, in his analysis of the problem for spherical particles, considered them to affect the settling both by their presence and by their motion. The introduction of a rigid particle into a fluid in motion, results in a distortion of the original field of flow since the fluid is reduced to rest at the particle surface in the absence of slipping. This effect for a number of spheres results in a decrease in the mean rate of shear between two parallel planes. Einstein's equation (1906) for the viscosity of a suspension obtained for this case:

$$\mu_c = \mu_o (1 + k c) \qquad (11)$$

where μ_c is the viscosity of the suspension, μ_o the viscosity of the pure liquid, c the volume concentration of solids and k is a numerical constant, described as a shape factor equal to 5/2 for rigid spheres. This expression is limited in application to dilute suspensions having a maximum concentration of about two per cent. At concentrations higher than this, mutual disturbances at the surfaces of the particles begin to exert their influence. Also, collisions are frequent, even at low concentrations.

For a settling suspension, what is required is the effective local resistance to shearing that would arise if there were a very small relative displacement between a given particle and the rest of

the suspension. The required expression was set forth by Vand (1948) as a special case in the theoretical treatment of the more complex case of the apparent bulk viscosity of a concentrated suspension. His equation is

$$\mu_c = \mu_o \exp (kc/(1 - Qc) \qquad (12)$$

where μ_c is now the effective local viscosity, Q is the interaction constant equal to 39/64 for rigid spheres and k is the usual 5/2.

Excluding the effect of the motion of the particles, Hawksley expressed the viscous drag on a given representative sphere in a suspension as if no other particles were present and as if the viscosity were μ_c as given in Equation 12. Hence, the drag in Stokes' expression $3\pi \mu_c U d_s$, where μ_c replaces μ_o, U equals the velocity and d_s is the diameter of the sphere.

Equation 12 does not represent the bulk viscosity that would be observed if the suspension were to be sheared in a viscometer, since only a negligibly small displacement is assumed in the derivation.

The effect of the motion of the particles upon settling was next considered. For Stokes' settling of a single sphere in an infinite fluid, the descending particle drags the entirety of the fluid along with it. This is impossible when the fluid is contained in a vessel and there is a return flow that produces a retarding effect which is unimportant unless the ratio of the diameter of the vessel to the diameter of the particle is insufficiently large. Due to this

"towing" effect, each particle in a suspension tends to drag down all other particles and a compact cloud of particles in a very large vessel will settle at rates exceeding the Stokes' velocity of the individual particles. If the cloud completely fills the vessel, however, the retarding effect of the return flow will outweigh the mutual downward drag and the actual settling rate is reduced to a value below the individual Stokes' velocity.

Thus, the representative sphere can be considered as settling in a pure fluid of viscosity as given by Equation 12 and subjected to a constant upward velocity. To evaluate this return velocity, u, an application of the continuity principle results in the expression

$$(1 - a)u + a U_c = 0 \qquad (13)$$

where U_c is the actual rate of settling of the spheres and a is the fraction of cross sectional area occupied by the spheres in an arbitrary horizontal plane. This fraction of solid area is easily shown to equal the volume concentration, c.

The return velocity, u, is then equal to $- c U_c/(1 - c)$. Since the actual velocity of settling, U_c, is equal to the algebraic sum of the Stokes' velocity, U, plus the return velocity, u,

$$U = U_c/(1 - c). \qquad (14)$$

From Equations 13 and 14, the viscous drag, D_c, on the representative

sphere, is determined as

$$D_c = \frac{3\pi \mu_o U_c d_s \exp(kc/1 - Qc)}{1 - c}. \qquad (15)$$

Inasmuch as the return flow concept entails the displacement of an equal volume of suspension upon the introduction of the representative sphere into the suspension, the density of the suspension, which is $cd_s + (1 - c) d_f$, determines the effective gravitational force acting upon the particle,

$$F_c = (1 - c)(d_s - d_f) g \pi D^3/6. \qquad (16)$$

Equating 15 and 16, the terminal velocity for the equilibrium settling of a suspension is obtained as

$$U_c = \frac{(d_s - d_f) g D^2}{18\mu_o}. \quad (1 - c)^2 \exp(- kc/1 - Qc) \qquad (17)$$

This cannot be expected to apply to flocculated systems or packed beds since in such cases, there are forces which prevent the establishing of the equilibrium arrangement. Also, this equation applies only to spherical particles, all of which are the same size. For a concentrated system containing a range of particle size (all spherical) there is little differential settling so long as the range is not too large, (e.g., five or six to one). The major difficulty in applying Equation

17 to such a system is the evaluation of D.

For non-spherical particles, the situation is more complex since the shape has an effect upon the viscous drag force and also upon the effective viscosity of the suspension. Hawskley's approach to the effect upon the viscous drag is to replace D in the Stokes' drag force by an equivalent diameter, D'. This diameter is that of a sphere having the same surface area as the irregular particle.

The effect of particle shape upon the effective viscosity was much simpler than the corresponding effect upon the bulk viscosity of the suspension since the rotation of the particles was assumed to be negligible. The correction for this case was applied to the constant k in Equation 17 resulting in

$$k = \frac{5}{2}\psi^{-3/2} \tag{18}$$

where ψ is the Wadell particle shape factor, which is commonly called the "sphericity".

Equation 17 was tested, utilizing data of Steinour (1944) for disperse systems of both spherical and irregular particles. The results were quite good for the more concentrated ranges of the suspensions. Some difficulties were experienced in obtaining reliable values of D' and ψ . Surprisingly, good agreement with theory over a wide range of concentrations was obtained with data on the sedimentation of red blood cells (non-rigid particles) dispersed in normal saline with sodium citrate utilizing a value of k in Equation 17 equal to that for spheres

(i.e., 2.5). The suggested explanation for this was that the increase in k due to the cell shape was balanced by a slight decrease due to the lack of rigidity of the cell.

All of the above approaches to the sedimentation phenomena of concentrated suspensions have held to a general viewpoint that as settling progressed, the porosity of the "actively settling portion" of the suspension remained constant. Also, a portion of the suspension that does not actively engage in the process is supposed to exist at the bottom of the settling chamber. This inactive zone is of zero height at the beginning of settling and gradually builds up as the top level of the suspension subsides. When the top level of the suspension and the top level of the inactive zone meet, the "critical" point is reached. Any additional settling is then due to the compression of this now totally inactive zone. An opinion quite different from this has been expressed in a recent monograph by Allison and Murray (1951). They view the settling of a suspension as the gradual decrease in the height of the suspension accompanied by a decrease in the porosity of the suspension. The porosity is assumed to be constant throughout the suspension at any given instant and all of the suspension is viewed as being "active". "Active" is used in the sense of presenting resistance to the flow of liquid. Utilizing this assumption, together with the equation

$$V = \frac{g\, d_{sus.}}{5\mu\, S^2} \cdot \frac{e^3}{(1-e)^2} \qquad (19)$$

(where V is the velocity of settling, g the acceleration due to gravity

and S is the specific surface of the powder) the authors were able to evaluate the specific surfaces (surface area per unit weight) of a number of powders. Their procedure was to obtain the customary sedimentation data of height of suspension at various values of time for dispersed suspensions of the powders. These data were plotted and graphically differentiated to obtain the velocity of fall of the surface of the suspension. For each value of velocity, corresponding values of the porosity, e, and the density of suspension, d_{sus}, were calculated, based upon the foregoing assumption. By plotting velocity versus the porosity function $e^3 d_{sus}/(1 - e)^2$, straight lines were obtained from whose slopes the values of the specific surface, S, were obtained. While the initial concentration affects the initial settling rate, the effect upon the calculated specific surface was found to give a maximum difference for the same material of 14 per cent. The surface areas so obtained were compared with results from air permeametric measurements, and nitrogen adsorption measurement. The agreement with the air permeametric figures was quite good. The nitrogen adsorption technique consistently gave higher values (as it also does in comparison with the air permeability results). The agreement found with the permeametric measurements would seem to lend some credence to Allison and Murray's fundamental concept of the nature of the sedimentation process.

CHAPTER III

EQUIPMENT AND EXPERIMENTAL PROCEDURE

In this investigation, methyl alcohol was used throughout as the suspending medium. It was found during a large number of preliminary tests in which water was used as the dispersing fluid that the suspended particles of various materials were often difficult to deflocculate and it was difficult to obtain consistent results upon aging the suspension. It is well known that settling rates are not only influenced by the usual variables of density, viscosity, particle size and concentration, but also by the surface nature of the particles themselves - i.e., whether or not the particles are hydrophilic or hydrophobic. As a suspending medium, methyl alcohol gave more consistent results than water for a wider variety of materials.

The equipment employed for obtaining the usual sedimentation data of subsidence of the level of the suspension as a function of time was quite simple. Graduated mixing cylinders of 100 ml. capacity were used as the settling chambers. These were of the glass stoppered variety and were Kimble's Exax Blue Line type. The mouth of each cylinder was about three-fourths the diameter of the main body of the cylinder. This width facilitated the insertion of powder into the cylinder.

The timer used was an electric pushbutton type obtained from

Fisher Scientific Company. It registered total elapsed time, indicating each tenth of a second and from 0.0 to 9999.9 seconds directly.

The methyl alcohol used throughout was of commercial purity (C.P.) and was manufactured by Merck and Company.

The permeameter that was used to obtain some of the specific surface areas of the powders consisted of a brass cylinder one inch in internal diameter into which the powder to be investigated was packed and through which dried air was drawn by a vacuum pump. The flow of air was downward through the bed of packed powder which was supported on a perforated brass disc, covered by an equal sized disc of coarse filter paper. A "U" tube manometer containing an oil having a specific gravity of 0.827 was connected across the cell to measure the drop in pressure of the air as it flowed through the bed of powder. Also, a Fisher and Porter Flowrator using tube number 08-150 with the required glass ball was employed to measure the quantity of air flowing through the bed.

This permeameter is a standard piece of equipment used in the Micromeritics Laboratory of The Georgia Institute of Technology and is described by DallaValle (1948).

In order to conduct a sedimentation experiment upon a powdered material, it was first necessary to determine what concentration range of the powder in the fluid would give the hindered or line type of settling. This was done by placing a known weight of the powder into a test tube (about one-third to one-half full) and then adding measured quantities of the fluid. After vigorous shaking, the suspension formed

was observed for evidences of hindered settling. More liquid was added until the first noticeable settling of the suspension (in a reasonable length of time) was obtained. This represented the upper concentration limit for hindered settling. More liquid was then added followed by shaking until the upper layer of the suspension became too diffuse for an accurate estimation of the height of the suspension (i.e., the line of separation between the clear supernatent liquid and the suspension became too indistinct). This concentration then represented the lower or most dilute limit for hindered settling. For dilutions beyond this, the suspension no longer settled in a body and the type of settling was no longer "hindered".

Having this approximate permissible concentration range, the weight of powder necessary to make up 100 ml. of the most dilute suspension that would give line settling was put into a mixing cylinder. Sufficient methyl alcohol was added (as long as the suspension formed was less concentrated than the maximum limit found previously) to bring the suspension level up to about 60 ml. A rubber stopper sealed the top of the cylinder. In this way, substantially the whole permissible concentration range could be covered in one cylinder.

The suspension was vigorously shaken to insure the complete dispersion of all of the powder. Due to the nature of the dispersing medium, there was no noticeable tendency for the particles to flocculate except for those powders which were extremely fine (e.g., talc and kaolin). Such materials were not investigated. Initially, a vacuum was applied to the space above the suspension after this shaking so as to evolve any trapped or adsorbed air that might be clinging to the

particles. Due to the high volatility of methanol and the disturbance of the suspension caused by the air and vapor ebulation, it was difficult to obtain an accurate zero time for the start of the settling process. It was found that a gentle rocking of the suspension to and fro in the cylinder following the vigorous shaking was sufficient to remove any entrapped air bubbles.

After the shaking and rocking, the cylinder was placed in an upright position and thereupon the timer was started. Readings of the volume of the suspension were taken at intervals during the course of the sedimentation until the volume remained essentially constant. The shaking and rocking was then repeated and a second check run was made. After the completion of this second run, the cylinder was put aside and allowed to remain undisturbed for 24 hours and the ultimate settled volume of the suspension was read.

The evidence for lack of flocculation was based on the relative difficulty of redispersing the sediment after it had remained undisturbed for such a long period of time. Had the suspension been flocculated, the sediment would have appeared as a "slime" and would have dispersed easily. Instead, a tight, closely packed bed was observed in all materials used in the correlations.

The suspension was then diluted with approximately 10 ml. of methyl alcohol and the whole settling process was then repeated. In this way, each material was tested at five concentrations giving a family of settling curves. Figure 1 shows the apparatus and the course of the experiment for a typical material.

The mixing cylinders employed were checked for uniformity of

4 Seconds

250 Seconds

500 Seconds

750 Seconds

Figure 1. The Hindered Setting of Silicon Carbide (400 Grit) in Methyl Alcohol (858 g./l.)

distance between the 10 ml. markings, throughout the length of the cylinder by means of a vernier caliper. It was necessary, however, to correct for one fault of manufacture that could have caused serious errors. In almost all of the cylinders used in these experiments, the barrels were not fixed on the bases so as to be perfectly vertical when the cylinders were placed on a horizontal plane. Even a slight deviation from verticality was sufficient to accelerate the settling by providing an easy path for the return flow of the fluid on the side opposite the direction of inclination. To correct for this, it was necessary to adjust the cylinder base. Perfect adjustment was obtained when there was no upward movement of suspension visible anywhere along the wall of the cylinder. This adjusting was performed prior to the taking of suspension volume readings. It was possible to place shims, marking the outline of the base upon them, and then lift the cylinder for shaking. The cylinder could then be replaced accurately upon the shims and the experiment commenced.

The specific gravity of the methyl alcohol was obtained from 75° to 90° F., the range of room temperature variation, by means of a sensitive hydrometer covering the range 0.7000 to 0.9000.

For the permeameter measurements, a known weight of powder was firmly packed by hand in the brass cell be means of a tight fitting plunger. The depth of the bed was obtained by the use of a depth gauge. Dried air was passed first through the flowmeter and then down through the compacted bed of powder. The pressure drop through the bed was then read upon the manometer which was connected across the cell. The vacuum pump drew upon a 12 liter reservoir flask that served as a

surge tank to remove fluctuations of pressure. This reservoir was connected to the permeameter cell. Several readings of pressure drop were taken at various flow rates. It was necessary for the pressure drop versus flow rate to plot as a straight line on log-log paper. This insured that only streamline flow was taking place within the bed.

CHAPTER IV

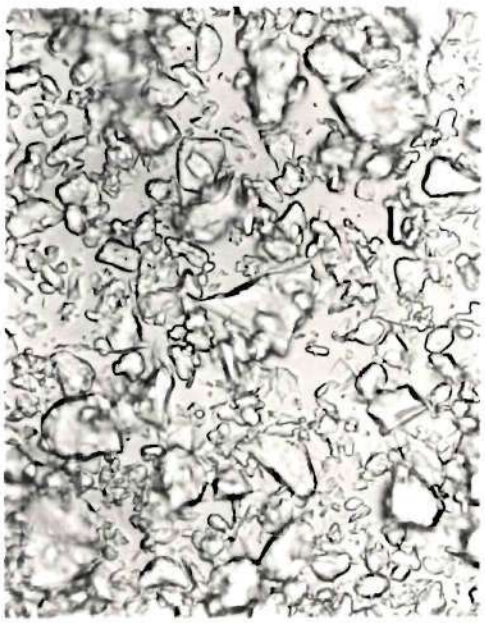
CORRELATION AND DISCUSSION OF RESULTS

Experimental Results.--The hindered settling of 17 different materials was observed. These materials were:

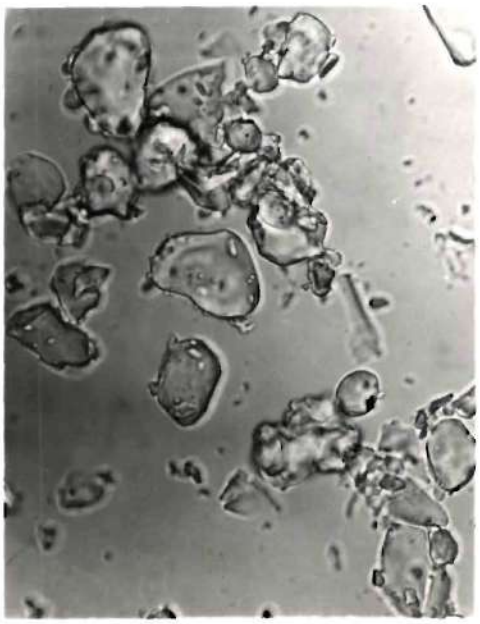
calcium carbonate	mercuric oxide
powdered zinc	silicon carbide (400 and 600)
powdered iron	calcium fluoride
powdered nickel	manganese dioxide
potassium sulfate	lead dioxide
potassium perchlorate	magnesium oxide
ferric oxide	aluminum oxide (levigated; 400 and 600 Grit).

Microphotographs of 12 of these are shown in Figures 2, 3 and 4.

Four of these materials were rejected from any consideration for reasons that appeared only after the respective experiments were completed. Lead dioxide, magnesium oxide and ferric oxide were discarded because of irregularities of behavior noticed along the walls of the settling cylinders. These materials tended to give a greater accumulation of sediment near the wall of the cylinders than in the mid-portion. This resulted in a buckled appearance of the surface of the suspension. Aside from making the reading of the volume of the suspension difficult, this signified that some sort of fluccolution and high surface forces were at work within the suspension. As the suspension became more concentrated with the passing of time, structures resembling miniature volcanoes appeared upon the upper surface of the suspension. These emitted little puffs of suspension into the clear supernatent liquid



$KClO_4$ (350X)



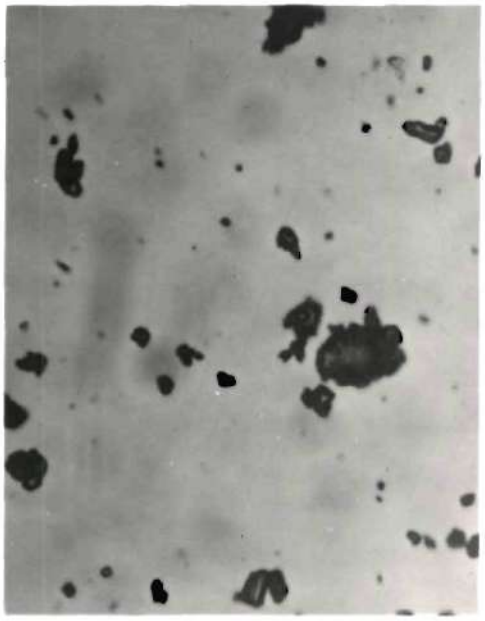
K_2SO_4 (350X)

SiC-400 (350X)

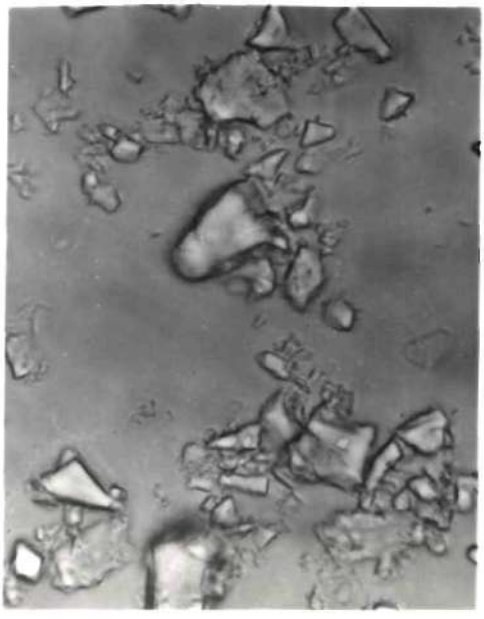
SiC-600 (1000X)

Figure 2. Microphotographs of Powders Investigated.

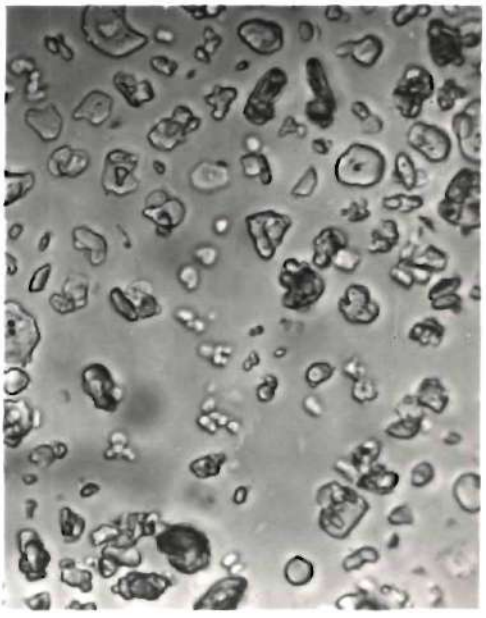
Ni (180X)



HgO (1000X)



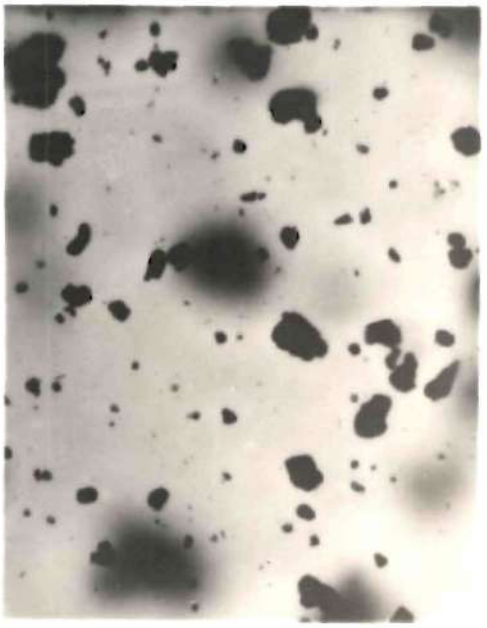
CaF_2 (1000X)



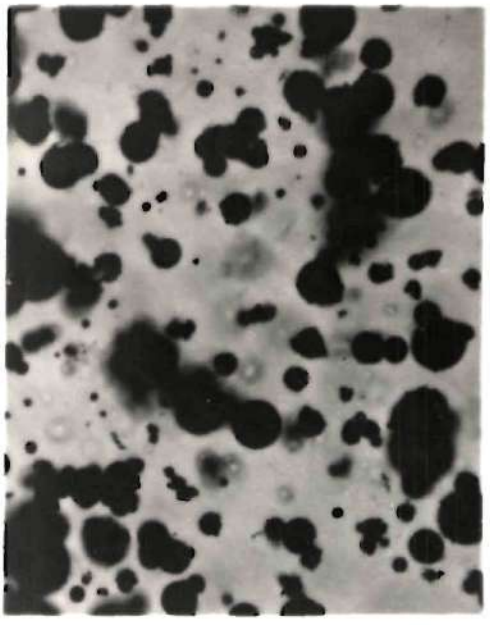
Al_2O_3 - lev. (1000X)

Figure 3. Microphotographs of Powders Investigated.

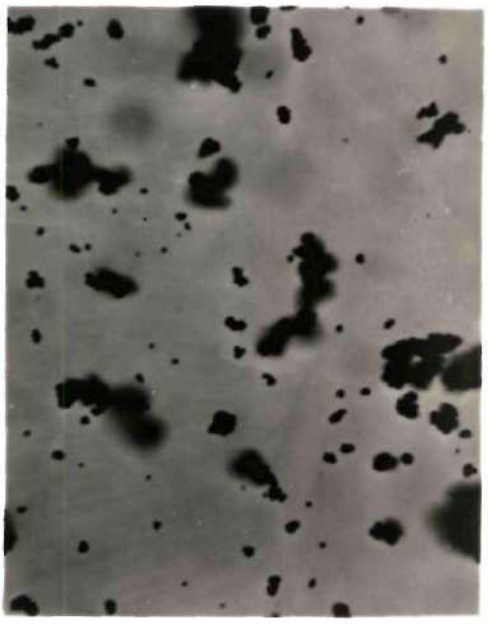
$CaCO_3$ (350X)



MnO_2 (1000X)



Zn (1000X)



Fe (1000X)

Figure 4. Microphotographs of Powders Investigated.

exactly as volcanoes emit smoke and lava. The occurrence of these volcanoes would seem to be due to the formation of fissures upon the advent of flocculation. These channels were widened and kept open by the return flow of the liquid being displaced during the concentration of the suspension. Therefore, settling was accelerated and the volume of the suspension was difficult to estimate.

This flocculation could have been avoided by the use of some surface active material to increase the wettability of these powders. This was not thought advisable since the effect of surface active agents upon the settling phenomena is still not certain. It is known that the ultimate settled height of a suspension is affected by such ingredients.

The 600 Grit aluminum oxide was also omitted from consideration as the powder contained a great deal of very fine material that prevented the settling from getting out, within a reasonable length of time, of the transient state mentioned by Hawksley (1950) as referred to in the Literature Survey. This was not due to the absolute size of this fine material but rather to the resulting wide range of particle size distribution. This is shown to be the case since the levigated alumina was very fine but settled quite nicely. Figure 10 in the appendix illustrates the fact that the transient nature was sustained for most of the 600 Grit settling. Figure 20 shows the normal behavior of the levigated alumina.

The family of curves that resulted for each type of material tested is shown in Figure 5 (for the five concentration of calcium carbonate in methyl alcohol). From this figure, it is seen that three

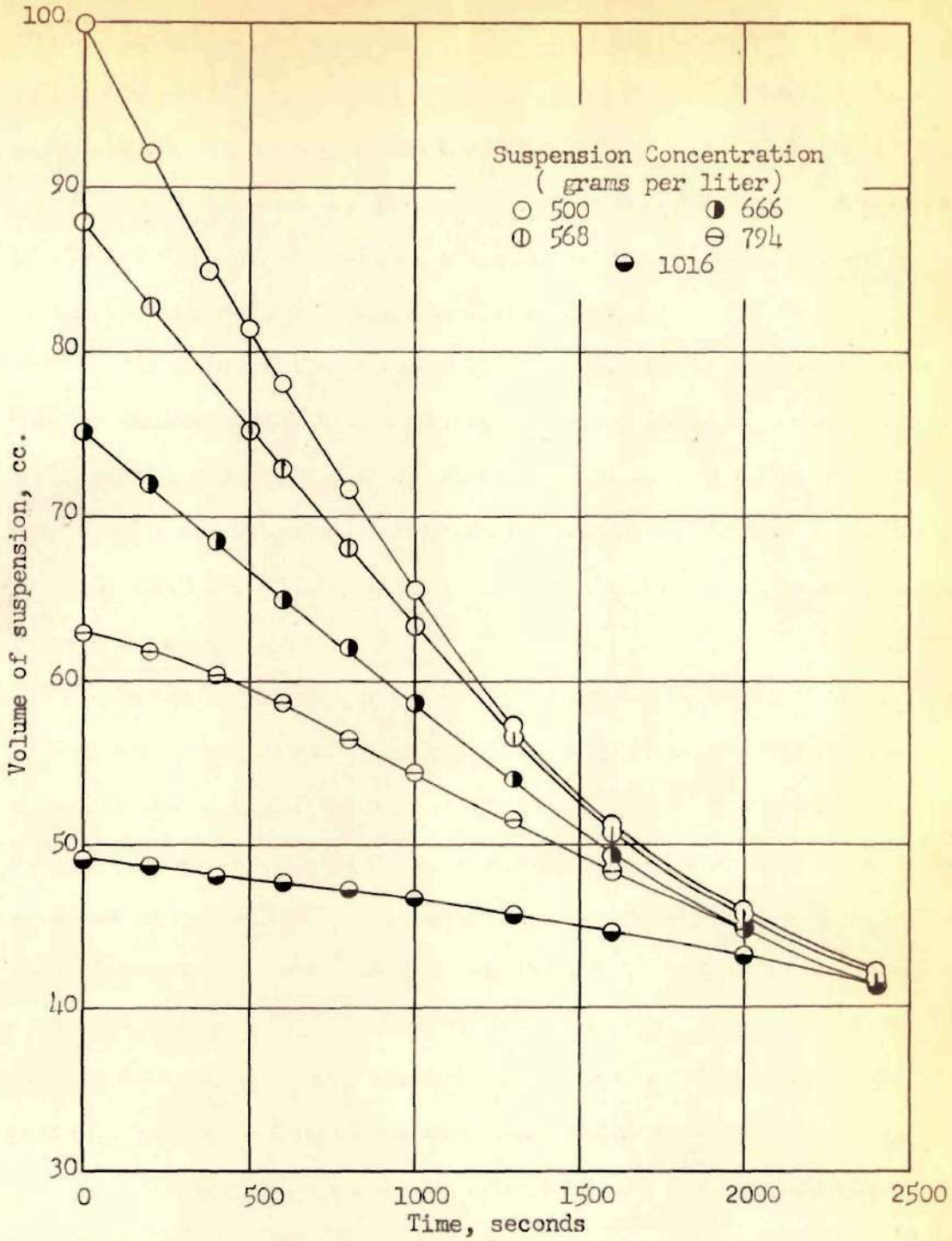


Figure 5. Subsidence of calcium carbonate suspension with time.

types of curves were obtained, depending upon the initial concentration of the suspension. The most dilute suspension is represented by the uppermost curve and the most concentrated by the lowest. Usually, for the most dilute suspension, the supernatent liquid was not completely clear but rather, carried a relatively small amount of the finer material that was present in the powder. However, the suspension level was clearly indicated. The two lower curves probably represent more the compression type of settling than the true hindered type. Therefore, it might be concluded that perhaps only two of this set of five curves truly represent that type of settling which is designated as "line" or "hindered".

Actually, plots of volume of suspension as a function of time have very little value except for the particular chamber in which the experiment was conducted. The reason for this is that the more significant dependent variable is actually the height of the suspension. To obtain the height of the suspension it is only necessary to divide by the internal cross sectional area of the cylinder. This is not usually necessary since the absolute height is not significant except for the particular cylinder employed. One significant quantity is a ratio of the suspension height at any time to the suspension height at infinite time. This is equal to the ratio of the respective volumes of the suspension. No internal area is necessary in this latter case in order to make the data of Figure 5 meaningful.

Since a ratio of volumes was seen to be necessary for any general correlation, several ratios were tried before a useful one was found. The volume function $V/(V_o - V)$ (equal to $H/(H_o - H)$, where V is the

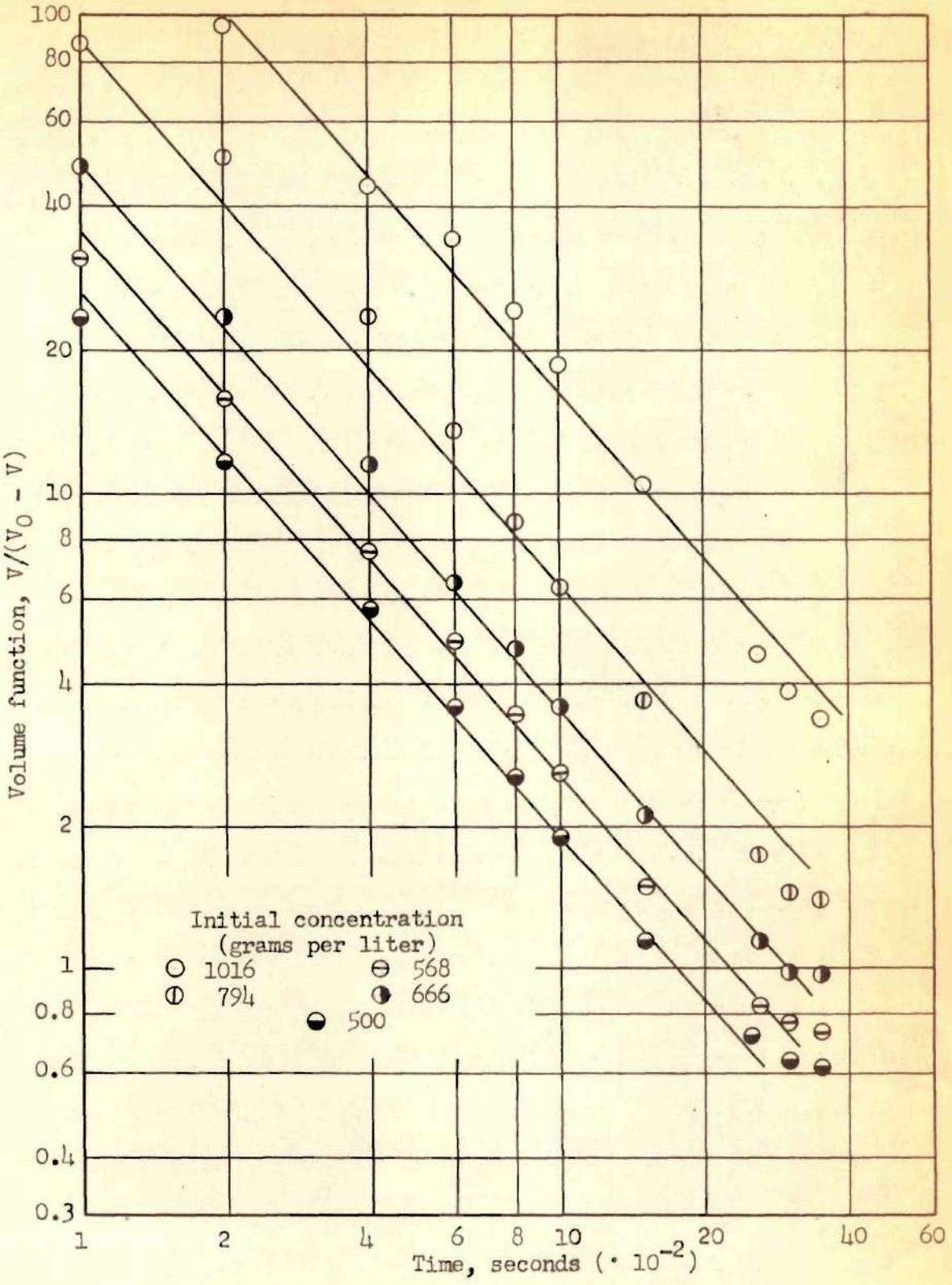


Figure 6. Volume function versus time for calcium carbonate

volume of suspension at any time and V_o is the initial volume of the suspension at zero time, was found to render the curves of Figure 5 into a series of points that could be approximated fairly well by a set of parallel straight lines. Figure 6 shows the resulting transformation for the same calcium carbonate family. Here the order of the curves has been reversed from that in Figure 5. The method used to obtain the parallel straight line was to select the data for a particular concentration, that could best be represented by a straight line, and then to place parallel lines through the data for the other concentrations as well as could be done by eye.

The upper two lines in Figure 6 correspond to the lower two in Figure 5. As was indicated above, these two did not represent a phenomenon similar to that observed in the other three curves. The least concentrated suspension is seen to have been in the hindered range also.

Deviations from the straight lines are noticed at both ends of the experimental date. (It is believed that these deviations represent firstly, the transient state (at the beginning of the experiment) and secondly, the obtaining of the compression zone near the end of the experiment). Of course, the curve of $V/(V_o - V)_v$ of versus time will become asymptotic to the value of the volume function with V equal to V_u, the ultimate settled volume of the suspension, as time increases.

The remaining experimental data was put into this form and is presented in the Appendix. It will be noticed that the data for several of the materials (alumina, 400 Grit; silicon carbide, 400 and 600 Grit) are well represented by the five sets of parallel straight lines. The

initial deviations are almost completely absent and the extremes in concentration are still well within the hindered range (using the adherence to a straight line as a criterion). These materials are all metallurgical polishing powders and appear to be closely sized. Particularly, very fine material was at a minimum. Contrary to that of the aluminum oxide, 600 Grit, mentioned earlier, the transient states would be of very short duration in these cases (orientation as Hawksley (1950) envisioned being easily accomplished when particles are nearly of uniform size).

The Correlation--Plotting the data on log-log paper in the form of the volume function $V/(V_o - V)$, versus time, t, one finds that an equation of the form

$$F(V) = \frac{V}{V_o - V} = A\ t^m \qquad (20)$$

where m is the slope of the parallel lines and A is a constant, represents the data. The slopes of the sets of parallel lines are presented in Table 1. The average value of the slope was found to be - 1.12.

From an inspection of the parallel lines, it was obvious that A was a function of the initial suspension concentration. A was calculated for each line by obtaining a value of F(V) at some convenient time and dividing this volume function by t^m. The m used here was the actual slope found for the particular line in question. A actually represents the value of F(V) at one second. Values of A are given in Table 2 for the various concentrations involved.

Table 1. Slopes of log $V/(V_o - V)$ Versus log t Curves

Material	Slope	Material	Slope
$CaCO_3$	- 1.17	CaF_2	- 1.19
Zn	- 1.15	MnO_2	- 1.08
H_gO	- 1.05	K_2SO_4	- 1.05
Fe	- 1.15	$KClO_4$	- 1.04
Ni	- 1.14	Al_2O_3 - lev.	- 1.19
SiC - 400	- 1.16	Al_2O_3 - 400	- 1.12
SiC - 600	- 1.05		
	Average Slope		- 1.12

Table 2. Intercepts of log $V/(V_o - V)$ Versus t Curves

Material and Concentration (g./l.)	Intercept A	Material and Concentration (g./l.)	Intercept A
Al_2O_3 - 600		K_2SO_4	
1385	4968	1351	3400
1160	3210	1148	1701
998	2140	1001	1058
875	1610	890	791
778	1272	801	605
		728	504
Al_2O_3, lev.		$KClO_4$	
850	32,700		
635	18,580	361	1094
533	12,300	306	682
460	9,290	268	427
401	7,140	238	288
SiC - 400		Ni	
1000	5160	2505	377
858	3845	2198	214
749	2813	1920	142
667	2175	1733	116
600	1788	1556	86
SiC - 600		$CaCO_3$	
600	10,460	1016	53,700
471	8,480	794	21,010
400	5,610	666	11,300
345	3,760	568	8,120
300	2,375	500	6,150

Table 2. Continued

Material and Concentration (g./l.)	Intercept A	Material and Concentration (g./l.)	Intercept A
HgO		Fe	
2866	1114	1141	2035
2510	815	1000	1198
2232	606	890	830
2008	431	800	633
Zn		CaF_2	
2320	6370	1208	40,500
1876	3977	1056	32,210
1559	2454	935	25,220
1351	1770	833	20,510
MnO_2			
681	607		
574	354		
500	246		
445	182		
400	145		

The values of A were plotted as functions of the initial concentration, C, on log-log paper, and the resulting straight lines are shown in Figure 7. These lines were drawn so that the greatest number would be parallel, i.e., given three or four materials that had straight parallel lines, the remaining lines were drawn so as to be parallel to these and yet represent as well as possible the actual data. Three materials (levigated alumina, powdered iron and powdered zinc) did not allow this to be done with much justification; therefore they are represented by their lines of unique slope. The remaining parallel straight lines are seen to fairly well represent the series of points. The slope of these parallel lines is 2.79. This signifies that the "constant" A is denoted by the equation

$$A = B\,C^{n} \tag{21}$$

where B is another "constant" and n is the slope of the lines and equal to 2.79.

The values of B were obtained as were the values of A above. Selected values of A were read from the lines at convenient values of C and the A's were divided by the respective values of $C^{2.79}$. B then represents the intercept of the A versus C line at C equal to one gram per liter.

An inspection of Figure 7 indicates that B itself is a function of some variable. It was felt that some index of the surface area which is active in retarding the flow of fluid through the suspension might be the desired variable. For this reason, surface areas obtained

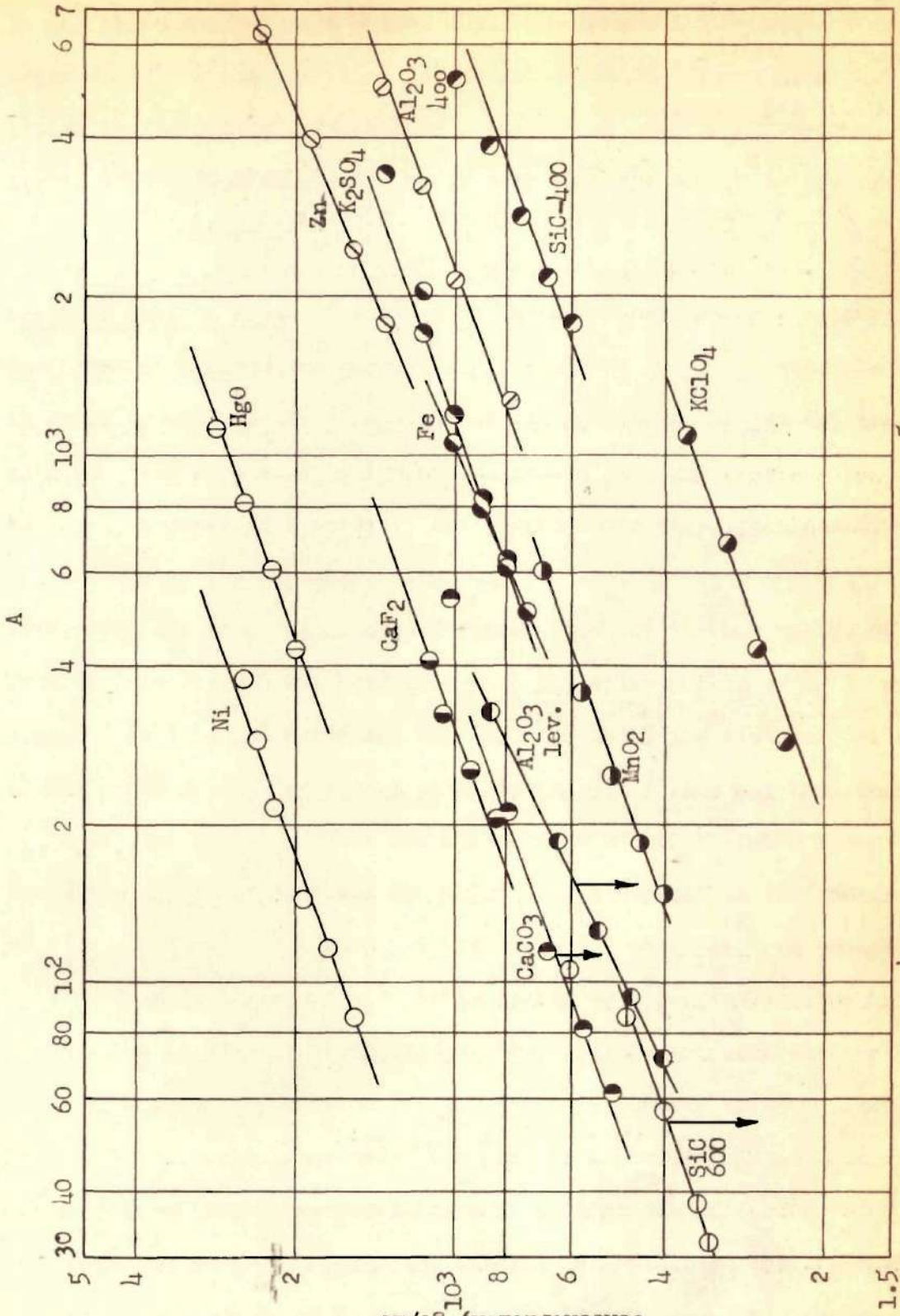


Figure 7. Plots of A versus concentration

by the gaseous adsorption method would not be applicable since those areas would include much more (of an interstitial nature) than that active in fluid flow. Since this interstitial area is often very large, the surface area would be grossly in error as far as the fluid problem is concerned.

Specific Surface Area--It was decided that the surface area measurement technique of Allison and Murray (1951) would be the most desirable since it actually employs the same fluid as the suspending medium and the data required were exactly those obtained in the experiments. For each material, a curve of V versus t was chosen whose slope continually changed during the experiment (usually the most dilute suspension). This curve was then graphically differentiated at various values of t. This differentiation was performed with the help of a mirror. It was placed normal to the curve and was adjusted until the visible portion of the curve became continuous with its image. A line was then drawn utilizing the bottom edge of the mirror as a straight edge. The cotangent of this normal was the value of the tangent to the curve (changed in sign). The slope of the curve was obtained from this tangent by multiplying by a suitable factor to take into account the differences in scale factors used on the abscissa and ordinate.

The porosity function $e^3 d_{sus.}/(1 - e)^2$ corresponding to each slope was then calculated and the slopes were plotted against the porosity functions. The resulting series of points was fitted to a straight line passing through the origin. A typical example of this is shown in Figure 8 for powdered nickel. As explained by Allison and

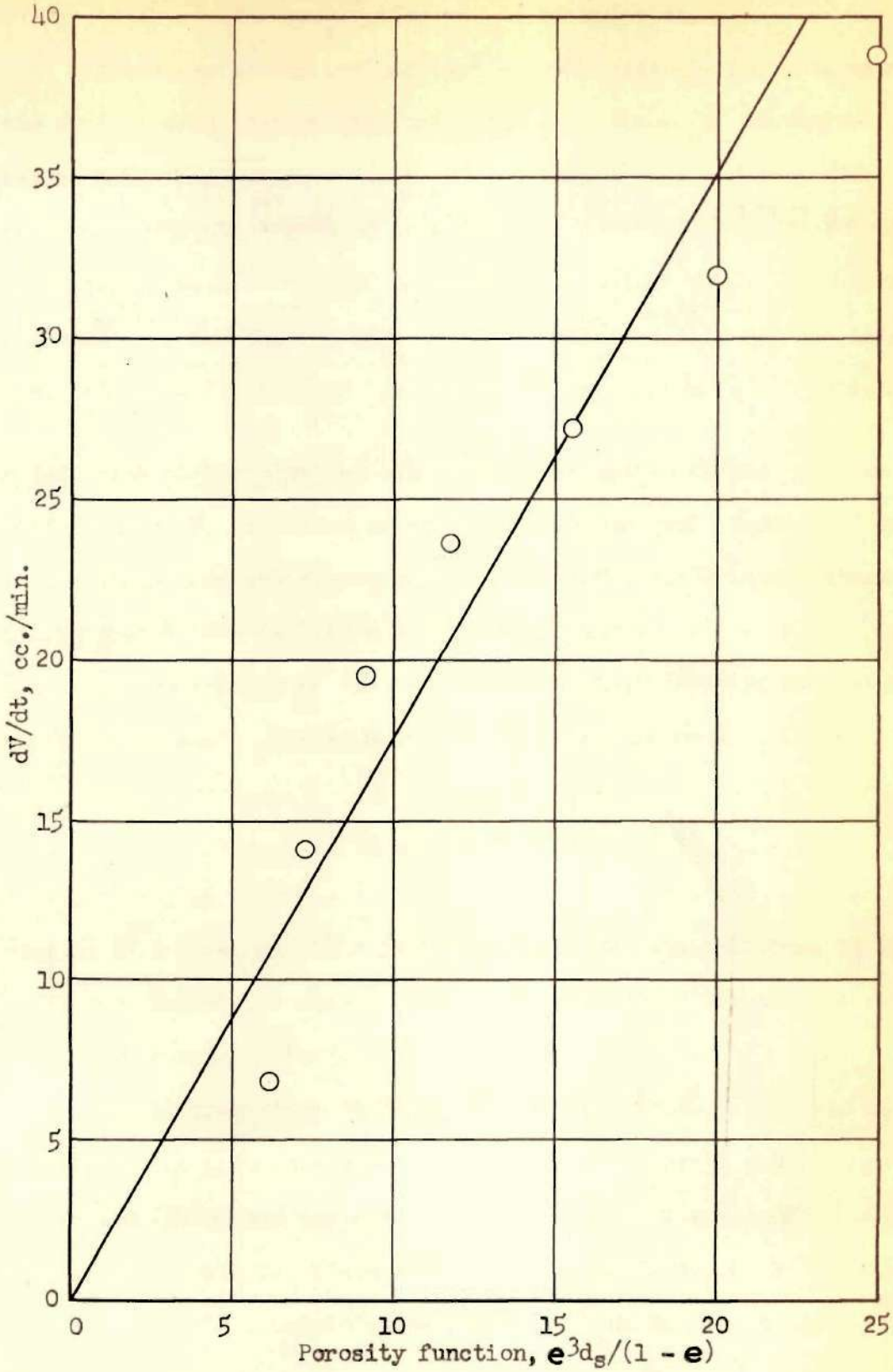


Figure 8. dV/dt versus porosity function for nickel

Murray, some scattering of points is to be expected.

The above method was not particularly suitable for determining the surface area of very fine materials (the slopes of the curves of V versus t in these cases usually changed very slowly and were difficult to obtain with any degree of accuracy). In these cases, the gas permeability method was used to obtain the surface area. This method was used only for the 600 Grit silicon carbide as this was the only very fine material carried through to the final step in the correlation.

Final State of Correlation.--The calculated values of the specific surface area, S, expressed as square meters per gram of material are listed along with the corresponding values of B in Table 3. These values are plotted in Figure 9 on log-log paper.

An examination of Figure 9 indicates that there is a clearly shown relationship between S and B. It takes the form

$$B = D\,S^{r} \tag{22}$$

where D is a constant and r is the slope of the straight line in Figure 9. By measurement, r equals 5.07 and D, calculated as were the other constants, equals 2.88.

The straight line in Figure 9 fits all of the data quite well except for the point designating potassium perchlorate. Professor J. M. DallaValle (1953) has indicated that this is an unusually difficult material for which to obtain a satisfactory value of the specific surface. The three remaining materials that do not appear in Figure 9

Table 3. Intercepts of log A Versus log Concentration Curves and Specific Surface Areas.

Material	Specific Surface S, m^2/g.	Intercept B ($.10^6$)
SiC - 600	0.1745	313.4
SiC - 400	0.1103	29.1
$KClO_4$	0.0615	82.1
K_2SO_4	0.0597	4.95
Al_2O_3 - 400	0.0997	9.76
HgO	0.0462	0.282
Ni	0.0296	0.113

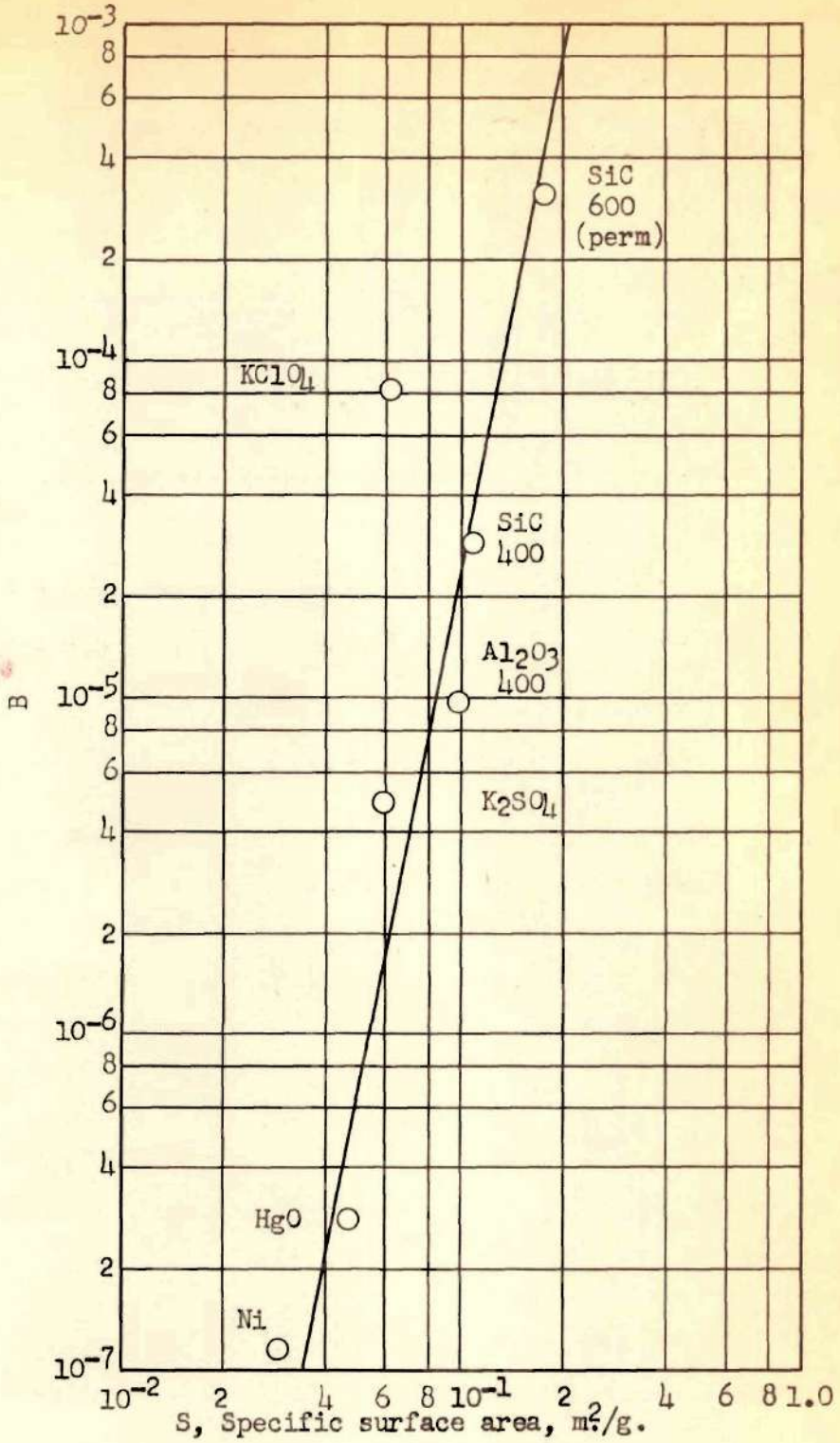


Figure 9. B versus the specific surface area, S

(calcium carbonate, manganese dioxide and calcium fluoride) were omitted for the same general reason.

Form of the Correlations.--The result of substituting the empirical values back into the progressive equations for the variable "constants" (A and B) gives the final form of the empirical equation for the hindered settling of powders in methyl alcohol at room temperature as

$$\frac{V}{V_o - V} = \frac{H}{H_o - H} = 2.88\, S^{5.07}\, C^{2.79}\, t^{-1.12} \qquad (23)$$

This equation has been tested for its ability to give back, upon substituting appropriate values for S, C and t, the experimental data obtained. The results of these calculations are presented in Table 4 which gives the per cent error of calculated values of V. In all cases (except alumina, 400 Grit) the values of t selected for the calculations were mid-range values, i.e., values of time at approximately the middle of the experiments. This selection was made in order to insure that only steady state hindered settling was occurring. In the case of alumina, 400 Grit, a mid-value of concentration was chosen and three values of time were used in the calculation. The values of S utilized are those given in Table 3.

The calculated results for the two silicon carbides perhaps best illustrate the trend of the errors. If one considered the amount of variation of the values of S from the straight line of Figure 6 and the number of such correlation steps, the errors, with two exceptions are

Table 4. Errors in Calculated Volume of Suspension

Material	Concentration g./l.	Time Sec.	V_{exp} cc.	$V_{calc.}$ cc.	Error %
H_gO	2866	100	62.7	64.4	≠ 2.7
$KClO_4$	601	100	69.2	39.4	- 43
	770	100	67.0	44.1	- 34
	1110	100	50.7	42.3	- 17
K_2SO_4	1001	100	71.8	55.9	- 22
	801	100	83.6	55.5	- 34
Al_2O_3 (400)	875	100	72.3	76.5	≠ 5.8
	875	200	65.3	72.6	≠ 11
	875	400	52.0	65.6	≠ 26
Ni	1733	20	48.1	39.4	- 18
	1733	50	35.3	24.5	- 30
SiC (400)	600	400	61.2	72.8	≠ 19
	667	400	60.1	70.4	≠ 17
	749	400	58.8	67.0	≠ 14
	858	400	55.3	61.5	≠ 11
	1000	400	50.2	55.0	≠ 9.7
SiC (600)	300	1000	62.9	58.5	- 7.0
	345	1000	62.6	59.9	- 4.3
	400	1000	60.0	57.0	- 5.0
	471	1000	54.2	53.0	- 2.1
	600	1000	43.2	45.4	≠ 2.8

small. The two exceptions mentioned are potassium perchlorate and potassium sulfate. It is felt that the large errors involved in these two materials are directly traceable to uncertainties in the values of the specific surfaces.

Undoubtedly, the use of Equation 23 to predict the hindered settling curves of powders is far outweighed by the possibility of using it to obtain a value of the specific surface area of a powder from its experimental behavior during hindered settling. It is to be observed that in Equation 23, a change in V (or H) will produce a much smaller change in S. An example of this is the calculation of S from the value of V_{exp} given in Table 4 for 400 Grit silicon carbide. A change of 18.9 per cent in the value of V resulted in a change of only 6.6 per cent in the calculated value of the specific surface area. It is hard to envision errors of such magnitude occurring during the conducting of a hindered settling experiment. This indicates that a powdered material can be subjected to a hindered sedimentation in methyl alcohol and the results of the experiment plotted in the manner of Figure 3. If this plot exhibits a straight line portion which has a slope approximating -1.12, Equation 23 can be used to obtain a value of S, the specific surface area of the powder.

This method has a decided advantage over the Allison and Murray technique for obtaining specific surface areas of powdered materials by virtue of its simplicity of calculation and lack of assumptions that might, in certain circumstances invalidate the latter method (e.g., applicability of Kozeny equation, excessive change in the value of the porosity of the suspension, inactivity of portions of the suspension,

etc.). It is to be noted that while the Allison and Murray method ignores the effect of concentration upon the specific surface area, Equation 23 gives a prominent place to this important variable.

An attempt was made to evolve some equations by dimensional analysis that would be applicable to hindered settling. Only two equations that seemed to have any significance resulted. They were

$$\left[\frac{g\ t}{\mu}\right] = F_1\left[H^4\ (d - d_o)\ S^4\ C^3\right] \tag{24}$$

and

$$\left[\frac{g\ t}{S\ \mu}\right] = F_2\left[H^4\ (d - d_o)\ S^4\ C^3\right] \tag{25}$$

where u is the velocity of fall of the suspension level, d_o is the density of the fluid and g is the acceleration of gravity - all in consistent units. Neither of these was able to reduce all of the data to a single curve. Other dimensionless groups were developed but they lacked any rational meaning.

CHAPTER V

CONCLUSIONS

It has been found by empirical methods that the hindered settling of alcohol suspensions of powders whose particles are roughly of irregular spherical shape can be represented at room temperature by the equation

$$\frac{V}{V_o - V} = \frac{H}{H_o - V} = 2.88\, S^{5.07}\, C^{2.79}\, t^{-1.12} \qquad (23)$$

within the experimental accuracy associated with the various quantities contained therein.

Through the use of Equation 23, the specific surface area of powdered materials, whose particles are roughly of irregular spherical shape and which form disperse suspensions in methyl alcohol, can be determined directly and simply by observing their hindered settling behavior at room temperature.

The specific surface area calculated from Equation 23 when appropriately employed as described above is at least as accurate as that obtained from either the gaseous permeability measurement or the Allison-Murray technique.

APPENDIX

Table 5. Data for Calcium Carbonate

Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$
1016	0	49.2		794	0	63.0		666	0	75.1	
	100	48.9	16.3		100	62.3	89.0		100	73.0	49.1
	200	48.7	97.5		200	61.8	50.6		200	72.0	23.2
	400	48.1	43.7		400	60.5	23.7		400	68.6	10.5
	600	47.8	34.2		600	58.7	13.5		600	65.1	6.5
	800	47.3	24.2		800	56.5	8.7		800	62.0	4.7
	1000	46.7	18.7		1000	54.5	6.4		1000	58.7	3.6
	1500	44.9	10.4		1500	49.5	3.7		1500	50.9	2.1
	2600	40.4	4.6		2600	40.0	1.7		2600	39.8	1.1
	3000	39.0	3.8		3000	37.3	1.5		3000	37.3	0.99
	3500	37.9	3.4		3500	36.8	1.4		3500	36.9	0.97
568	0	88.0		500	0	100.0					
	100	85.3	31.0		100	95.9	23.4				
	200	82.8	15.8		200	92.1	11.7				
	400	77.8	7.6		400	85.0	5.6				
	600	73.0	4.9		600	78.2	3.6				
	800	68.2	3.4		800	71.7	2.5				
	1000	63.5	2.6		1000	65.6	1.9				
	1500	52.4	1.5		1500	53.0	1.1				
	2600	40.2	0.84		2500	41.6	0.713				
	3000	38.1	0.76		3000	39.0	0.64				
	3500	37.1	0.73		3500	37.9	0.61				

Table 6. Data for Alumina - 600 Grit

Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$
998	0	50.1		830	0	60.2		715	0	70.0	
	200	49.5	76.1		200	59.3	65.9		200	69.0	69.0
	300	49.3	61.6		300	59.0	49.2		400	68.1	35.8
	500	49.0	44.5		500	58.6	35.5		600	67.4	25.9
	700	48.8	37.5		700	58.0	26.4		800	66.9	21.2
	1000	48.2	25.4		1000	57.3	19.7		1000	66.0	16.5
	1600	47.1	15.7		1600	56.0	13.3		1600	64.2	11.1
	2000	46.4	12.5		2000	55.0	10.6		2000	63.0	9.0
	2600	45.4	9.5		2600	53.4	7.9		2600	61.0	6.8
	3000	44.8	8.5		3000	52.4	6.7		3000	59.6	5.7
624	0	80.2		555	0	90.1					
	200	79.0	65.8		200	89.0	80.9				
	400	78.0	35.4		400	88.0	41.9				
	600	77.1	24.9		600	86.9	27.2				
	800	76.3	19.1		800	85.8	19.9				
	1000	75.4	15.7		1000	84.7	15.7				
	1600	72.9	10.0		1600	81.4	9.4				
	2000	71.1	7.8		2000	79.2	7.2				
	2400	69.3	6.3		2400	77.0	5.9				
	3000	66.7	5.0		3000	73.8	4.5				
	4000	62.0	3.4		4000	68.0	3.1				

Table 7. Data for Alumina - 400 Grit

Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$
1385	0	50.5		1160	0	60.2		998	0	70.1	
	100	48.7	27.1		100	57.0	17.8		100	64.9	12.5
	200	47.0	13.4		200	54.0	8.7		150	62.4	8.0
	300	45.1	8.4		300	50.8	5.4		200	60.0	5.9
	400	43.3	6.0		400	48.0	3.9		300	55.1	3.7
	500	42.4	5.2		500	45.0	3.0		400	50.4	2.5
	600	42.3	5.2		600	43.0	2.5		500	46.0	1.9
	700	42.2	5.1		700	43.0	2.5		600	43.2	1.6
									700	43.2	1.6
875	0	80.0		778	0	90.0					
	100	72.3	9.4		100	80.1	8.0				
	150	69.0	6.3		150	75.3	5.1				
	200	65.3	4.4		200	70.6	3.6				
	300	58.7	2.8		300	61.2	2.1				
	400	52.0	1.9		400	52.5	1.4				
	500	46.1	1.4		500	46.1	1.1				
	600	43.0	1.2		600	43.1	0.92				
	700	43.0	1.2		700	43.0	0.92				

Table 8. Data for Powdered Nickel

Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$
2505	0	42.3		2198	0	48.2		1920	0	55.2	
	10	40.4	21.2		10	45.0	14.1		10	49.3	8.4
	15	39.8	15.9		15	43.7	9.6		15	47.0	5.8
	20	39.1	12.0		20	42.2	7.0		20	45.0	4.4
	30	37.7	8.2		30	39.4	4.5		30	41.3	3.0
	40	36.2	5.9		40	36.9	3.3		40	37.7	2.2
	50	34.6	4.5		50	34.5	2.5		50	34.4	1.7
	60	33.0	3.5		70	30.5	1.7		60	32.0	1.4
	70	31.1	2.8		85	29.8	1.6		70	30.3	1.2
	90	29.2	2.2						90	29.7	1.2
1733	0	61.1		1556	0	68.2					
	10	54.2	7.9		10	56.6	4.9				
	20	48.1	3.7		20	49.3	2.6				
	30	43.8	2.5		30	43.4	1.8				
	40	39.0	1.8		40	38.5	1.3				
	50	35.3	1.4		50	34.5	1.0				
	60	32.7	1.2		60	32.1	0.89				
	70	31.0	1.0		70	30.5	0.81				
	80	30.1	0.97		80	30.0	0.79				
	90	30.0	0.96		90	29.9	0.78				

Table 9. Data for Silicon Carbide - 400 Grit

Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$
1000	0	60.0		858	0	70.0		749	0	80.5	
	100	57.7	25.1		100	66.2	17.2		100	74.9	13.4
	200	55.3	11.6		150	64.4	11.5		150	72.2	8.7
	250	54.0	9.0		200	62.7	8.6		200	69.6	6.5
	300	52.8	7.3		250	60.9	6.7		250	66.9	4.9
	400	50.2	5.1		300	59.0	5.4		300	64.1	3.9
	500	47.8	3.9		400	55.3	3.8		400	58.8	2.7
	600	46.5	3.4		500	51.8	2.8		500	53.6	2.0
	700	46.4	3.4		600	48.1	2.2		600	49.0	1.6
	800	46.4	3.4		700	46.8	2.0		700	46.7	1.4
					800	46.8	2.0		800	46.7	1.4
667	0	90.0		600	0	100.0					
	100	82.1	10.3		100	89.7	8.7				
	150	78.3	6.7		150	84.7	5.5				
	200	74.6	4.8		200	79.7	3.9				
	250	70.8	3.7		250	74.8	3.0				
	300	67.1	2.9		300	69.9	2.4				
	400	60.1	2.0		400	61.2	1.6				
	500	53.9	1.5		500	54.4	1.2				
	600	49.0	1.2		600	49.4	0.98				
	700	46.3	1.1		700	46.5	0.87				
	800	46.3	1.1		800	46.4	0.87				

Table 10. Data for Silicon Carbide - 600 Grit

Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$
600	0	50.0		471	0	63.7		400	0	75.0	
	100	49.4	82.4		100	62.8	69.8		100	73.3	43.1
	200	48.9	42.5		200	62.0	35.4		200	71.7	21.7
	300	48.2	26.0		300	61.0	22.2		300	70.1	14.2
	400	47.6	19.4		400	60.0	16.0		400	68.7	10.9
	500	46.9	15.1		500	59.0	12.6		500	67.2	8.6
	700	45.3	9.6		700	57.0	8.5		700	64.5	6.1
	1000	43.2	6.4		1000	54.2	5.67		1000	60.0	4.0
	1500	39.5	3.8		1500	49.2	3.4		1500	52.9	2.4
	2000	33.9	2.1		2000	44.1	2.2		2000	45.9	1.6
	3000	30.9	1.6		3000	32.9	1.1		3000	33.8	0.82
					4000	30.0	0.89		4100	29.9	0.66
345	0	87.0		300	0	100.0					
	100	84.0	28.0		100	95.1	19.4				
	200	81.4	14.5		200	90.3	9.3				
	300	78.9	9.7		300	85.8	6.1				
	400	76.3	7.1		400	81.1	4.3				
	500	74.0	5.7		500	77.0	3.4				
	700	69.1	3.85		700	70.0	2.3				
	1000	62.6	2.6		1000	62.9	1.7				
	1500	54.0	1.6		1500	52.9	1.1				
	2000	46.5	1.1		2000	46.9	0.89				
	3000	34.4	0.65		3000	34.7	0.53				
	4000	29.8	0.52								

Table 11. Data for Potassium Perchlorate

Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$
1110	0	54.0		910	0	66.0		770	0	78.0	
	10	52.9	48.1		10	65.1	72.4		10	76.5	49.3
	20	52.6	36.2		20	64.6	46.1		20	75.3	27.8
	30	52.3	30.8		30	64.0	31.2		30	74.3	20.1
	50	51.9	24.7		50	62.9	20.0		50	72.1	12.1
	70	51.3	19.0		70	61.6	14.0		70	70.0	8.8
	100	50.7	15.1		100	59.8	9.7		100	67.0	6.1
	150	49.4	10.7		150	56.8	6.2		150	61.6	3.8
	200	48.3	8.5		200	53.9	4.5		200	56.1	2.6
	250	47.5	7.3		250	51.0	3.4		250	51.3	1.9
	300	46.7	6.4		300	48.8	2.8		300	48.6	1.7
	400	45.9	5.7		400	46.6	2.4		400	46.3	1.5
675	0	89.0		601	0	99.9					
	10	86.4	32.6		10	96.3	26.4				
	20	84.5	18.6		20	92.9	13.2				
	30	82.7	13.0		30	89.7	8.8				
	50	79.0	7.9		50	83.9	5.2				
	70	75.4	5.5		70	77.9	3.5				
	100	69.9	3.7		100	69.2	2.3				
	150	60.8	2.2		150	57.9	1.4				
	200	54.0	1.5		200	52.6	1.1				
	250	50.4	1.3		250	50.2	1.0				
	300	48.8	1.2		300	48.8	0.96				
	400	47.8	1.2		400	48.0	0.93				

Table 12. Data for Potassium Sulfate

Conc. g./e.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./e.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./e.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$
1351	0	59.2		1148	0	69.8		1001	0	79.9	
	20	58.7	130.6		20	68.5	54.8		20	78.0	42.1
	40	58.3	68.6		40	67.8	33.9		40	76.2	20.9
	60	58.0	48.3		60	66.9	23.5		60	74.8	14.8
	80	57.4	32.8		80	66.0	17.6		80	73.2	11.0
	100	57.0	26.5		100	65.0	13.7		100	71.8	8.9
	160	55.9	17.2		160	62.4	8.5		160	67.3	5.4
	260	53.8	10.1		260	57.9	4.9		260	60.2	3.1
	400	50.6	5.9		400	51.7	2.9		400	52.4	1.9
	500	49.0	4.8		500	49.7	2.5		500	50.4	1.7
890	0	90.0		801	0	99.9		728	0	109.9	
	20	87.1	30.0		20	96.0	24.3		60	96.8	7.4
	40	84.9	16.6		40	92.6	12.6		80	92.7	5.4
	60	82.6	11.2		60	89.5	8.6		100	88.7	4.2
	80	80.5	8.5		80	86.5	6.5		160	77.2	2.4
	100	78.4	6.8		100	83.6	5.1		260	63.0	1.3
	160	72.2	4.1		160	74.9	3.0		400	54.9	1.0
	260	62.0	2.2		260	62.1	1.6		500	53.0	0.93
	400	53.6	1.5		400	54.0	1.2				
	500	51.6	1.3		500	52.0	1.1				

Table 13. Data for Mercuric Oxide (Red Precipitate)

Conc. g./e.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./e.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./e.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$
3338	0	60.0		2866	0	69.8		2510	0	79.8	
	10	58.7	43.4		10	68.8	68.8		10	78.5	62.8
	20	58.3	34.3		20	68.1	40.1		20	77.4	33.0
	30	57.9	27.6		30	67.4	28.1		30	76.4	22.4
	40	57.5	22.5		40	66.8	22.3		40	75.3	16.9
	50	57.1	19.3		50	66.0	17.4		50	74.3	13.6
	70	56.3	15.2		70	64.7	12.7		70	72.1	9.4
	100	55.1	11.1		100	62.7	8.8		100	69.0	6.4
	150	54.1	9.1		150	59.2	5.6		150	63.6	3.9
					200	55.9	4.0		200	58.2	2.7
									300	55.5	2.3
2232	0	89.7		2008	0	99.7					
	10	88.0	51.7		10	96.8	33.4				
	20	86.4	26.2		20	94.4	17.6				
	30	84.7	17.1		30	92.1	12.0				
	40	83.0	12.5		40	89.8	9.1				
	50	81.5	9.9		50	87.6	7.3				
	70	78.3	6.9		70	83.1	5.0				
	100	73.3	4.5		100	76.6	3.3				
	150	65.2	2.7		150	66.2	2.0				
	200	57.5	1.8		200	57.8	1.4				
	300	55.1	1.6		300	54.5	1.2				

Table 14. Data for Manganese Dioxide

Conc. g./l.	Time Sec.	Volume of Suspension	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension	$\frac{V}{V_o - V}$
681	0	58.7		574	0	69.8		500	0	80.0	
	100	57.5	47.9		100	67.2	25.4		100	76.2	19.8
	150	57.0	33.6		150	66.1	17.9		150	74.5	13.6
	200	56.5	25.1		200	65.1	13.7		200	72.8	10.1
	300	55.3	16.3		300	63.0	9.3		300	69.4	6.5
	400	54.1	11.6		400	61.0	6.9		400	66.0	4.7
	500	53.0	9.3		500	59.0	5.5		500	62.6	3.6
	700	50.5	6.2		700	55.0	3.7		700	56.1	2.4
	1000	47.0	4.0		1000	49.0	2.4		1000	48.4	1.5
	1500	43.1	2.8		1500	43.3	1.6		1500	43.4	1.2
445	0	90.0		400	0	99.9		364	0	109.9	
	100	84.6	15.7		100	93.0	13.5		150	96.8	7.4
	150	82.0	10.3		150	89.5	8.6		200	92.6	5.4
	200	79.4	7.5		200	86.1	6.2		300	84.0	3.2
	300	74.7	4.9		300	79.5	3.9		400	75.8	2.2
	400	69.7	3.4		400	73.0	2.7		500	67.9	1.6
	500	65.0	2.6		500	66.5	2.0		700	56.2	1.1
	700	56.3	1.7		700	56.2	1.3		1000	48.2	0.78
	1000	48.3	1.2		1000	48.3	0.94		1500	43.6	0.66
	1500	43.5	0.77		1500	43.7	0.78				

Table 15. Data for Iron (Hydrogen Reduced)

Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$
1363	0	58.7		1141	0	70.0		1000	0	80.0	
	20	58.0	89.2		20	68.6	47.3		20	77.9	37.1
	40	57.6	52.3		40	67.8	30.2		40	75.7	17.6
	60	57.1	36.8		60	66.9	21.6		60	73.3	10.9
	80	56.8	30.7		80	65.4	14.2		80	71.0	7.9
	100	56.4	25.1		100	64.0	10.6		100	68.7	6.1
	160	55.2	16.0		160	59.6	5.7		160	61.6	3.3
	200	54.3	12.5		200	57.0	4.4		200	57.0	2.5
	300	51.1	6.7		300	51.3	2.7		300	51.7	2.0
	400	49.4	5.3		400	51.3	2.7		400	51.7	2.0
890	0	90.0		800	0	100.0					
	20	86.4	24.0		20	95.0	19.0				
	40	83.1	12.0		40	90.4	9.4				
	60	79.9	7.9		60	86.0	6.1				
	80	76.6	5.7		80	81.6	4.4				
	100	73.5	4.4		100	77.2	3.4				
	160	63.8	2.4		160	64.3	1.8				
	200	57.7	1.8		200	57.6	1.4				
	300	52.0	1.4		300	52.0	1.1				
	400	52.0	1.4		400	52.0	1.1				

Table 16. Data for Powdered Zinc

Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$
2320	0	51.8		1876	0	64.0		1559	0	77.1	
	100	50.0	27.8		100	60.6	17.8		100	71.0	11.6
	200	48.2	13.4		200	57.7	9.2		200	65.7	5.7
	300	46.7	9.2		300	54.8	6.0		300	60.1	3.5
	500	43.3	5.1		500	48.7	3.2		500	49.9	1.8
	700	40.0	3.4		700	43.0	2.1		700	42.6	1.2
	900	37.0	2.5		900	38.6	1.5		900	38.0	0.96
	1000	35.3	2.1		1000	37.0	1.4		1000	36.5	0.90
	1500	33.2	1.8		1500	33.0	1.1		1500	32.9	0.74
1351	0	88.9									
	100	79.9	8.9								
	200	71.9	4.2								
	300	63.8	2.5								
	500	49.5	1.3								
	700	41.9	0.89								
	900	37.5	0.73								
	1000	36.0	0.68								
	1500	32.9	0.59								

Table 17. Data for Levigated Alumina

Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$
850	0	47.0		635	0	62.9		533	0	75.0	
	100	46.1	51.2		100	61.8	56.2		100	73.0	36.5
	300	45.5	29.3		300	59.9	19.6		300	69.9	13.7
	500	44.8	20.4		500	57.8	11.3		500	66.7	8.0
	700	43.9	13.9		700	55.9	7.9		700	63.4	5.5
	900	42.9	10.5		900	53.8	5.9		900	60.3	4.1
	1300	41.0	7.8		1100	51.9	4.7		1100	57.1	3.2
	1700	39.1	4.9		1300	49.9	3.8		1300	54.1	2.6
	2100	37.2	3.8		1500	47.9	3.2		1500	51.2	2.1
	3000	32.3	2.2		1700	45.9	2.7		1700	48.3	1.8
					2100	42.0	2.1		2100	43.2	1.4
					3000	33.9	1.2		3000	34.2	0.84
460	0	87.0		401	0	99.8					
	100	84.4	31.8		100	95.8	23.9				
	300	79.5	10.6		300	88.4	7.7				
	500	74.7	6.1		500	81.2	4.4				
	700	69.9	4.1		700	74.3	2.9				
	900	65.0	3.0		900	67.6	2.1				
	1100	60.4	2.3		1100	61.7	1.6				
	1300	56.0	1.8		1300	56.7	1.3				
	1500	52.2	1.5		1500	52.6	1.1				
	1700	49.0	1.3		1700	49.1	0.97				
	2100	43.4	1.0		2200	42.4	0.74				
	3000	34.4	0.65		3000	34.4	0.53				

Table 18. Data for Calcium Fluoride

Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$	Conc. g./l.	Time Sec.	Volume of Suspension cc.	$\frac{V}{V_o - V}$
1208	0	62.1		1056	0	71.0		935	0	80.3	
	100	61.7	176.3		100	70.2	87.8		100	79.0	63.2
	200	61.2	68.0		200	69.6	49.7		200	78.0	34.7
	300	60.7	45.0		300	68.9	32.8		300	77.1	24.1
	500	59.7	25.4		500	67.6	19.6		500	75.2	14.9
	700	58.5	16.5		700	66.1	13.3		700	73.4	10.7
	1000	57.0	11.2		1000	64.1	9.2		1000	70.7	7.41
	1500	54.1	6.8		1500	60.5	5.8		1500	65.8	4.6
	2600	48.4	3.5		2600	52.0	2.7		2600	54.1	2.1
	3600	43.5	2.3		3600	46.6	1.9		3600	48.0	1.5
833	0	90.2									
	100	88.6	57.1								
	200	87.3	30.6								
	300	86.1	21.0								
	500	83.9	13.3								
	700	81.5	9.4								
	1000	77.9	6.3								
	1500	71.8	3.9								
	2600	58.0	1.8								
	3600	50.0	1.3								

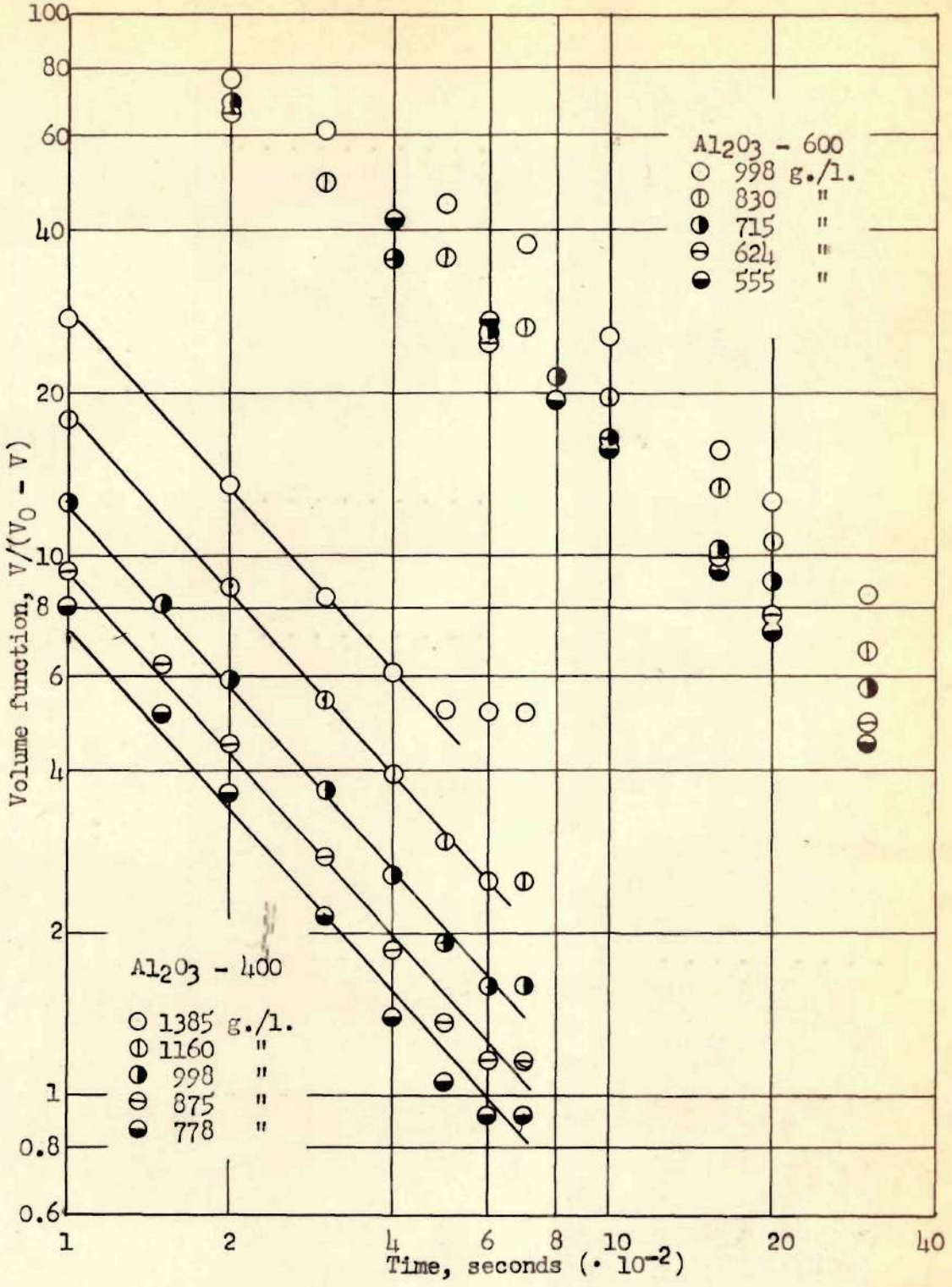


Figure 10. Volume function versus time for the aluminas

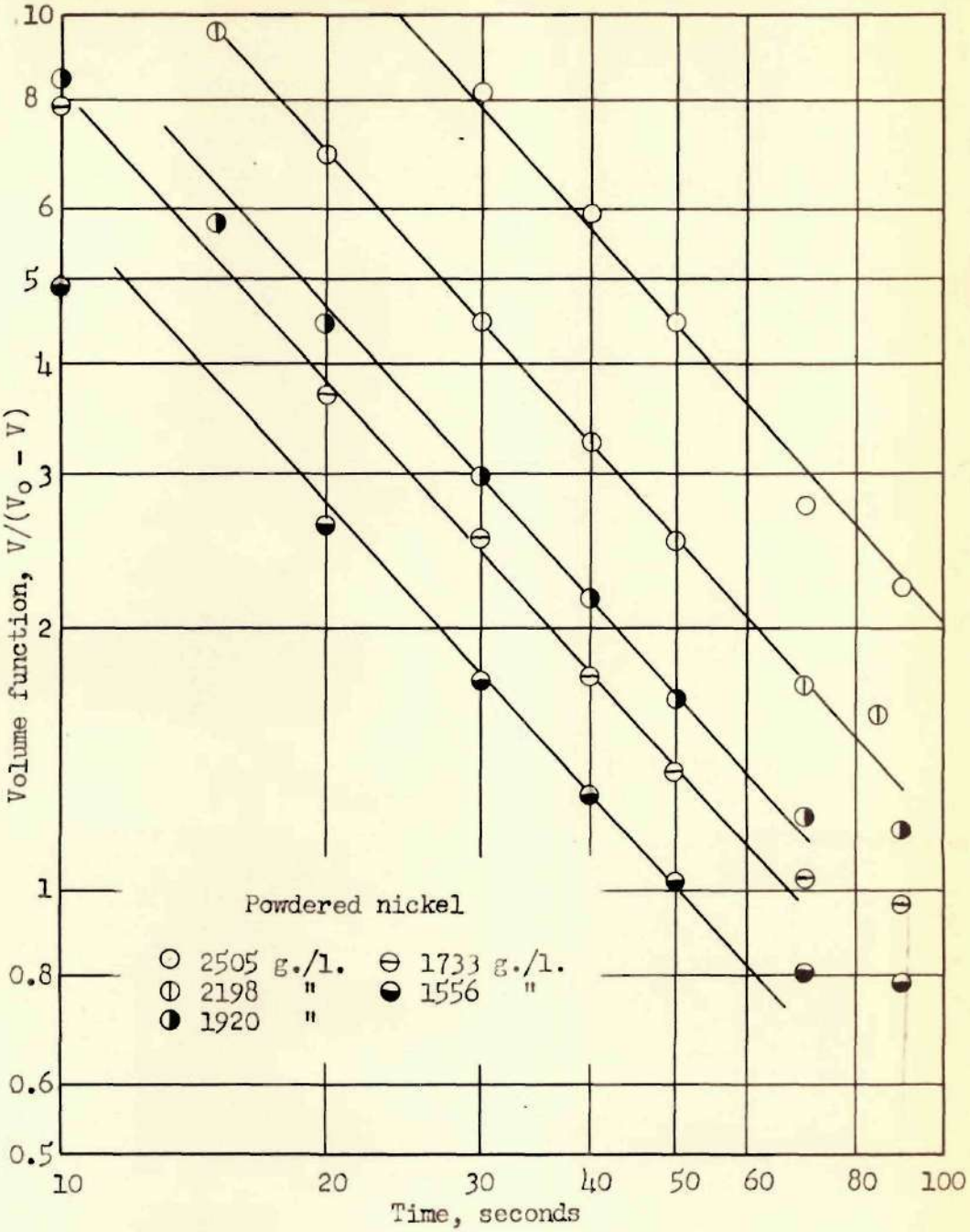


Figure 11. Volume function versus time for powdered nickel

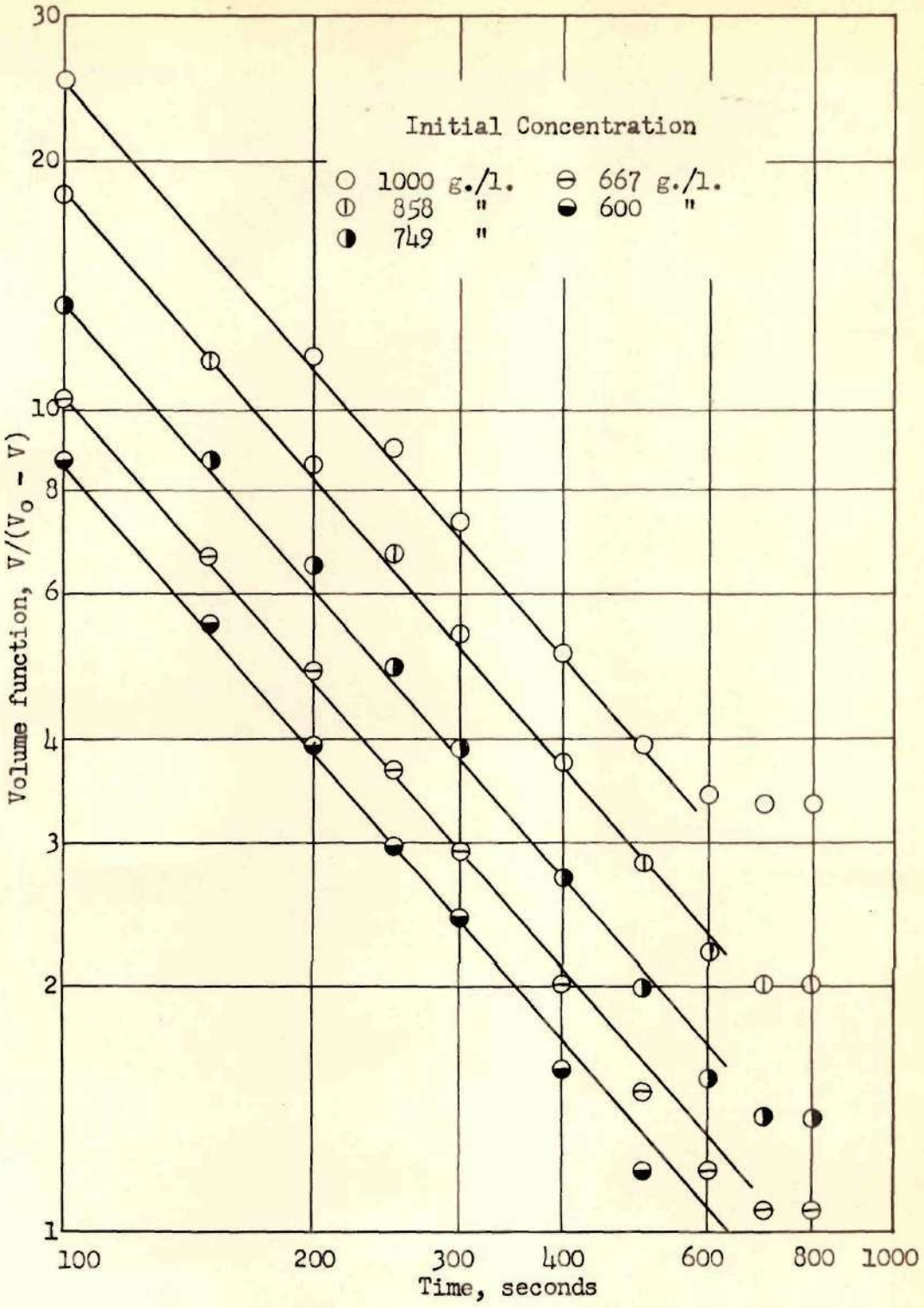


Figure 12. Volume function versus time for silicon carbide - 400

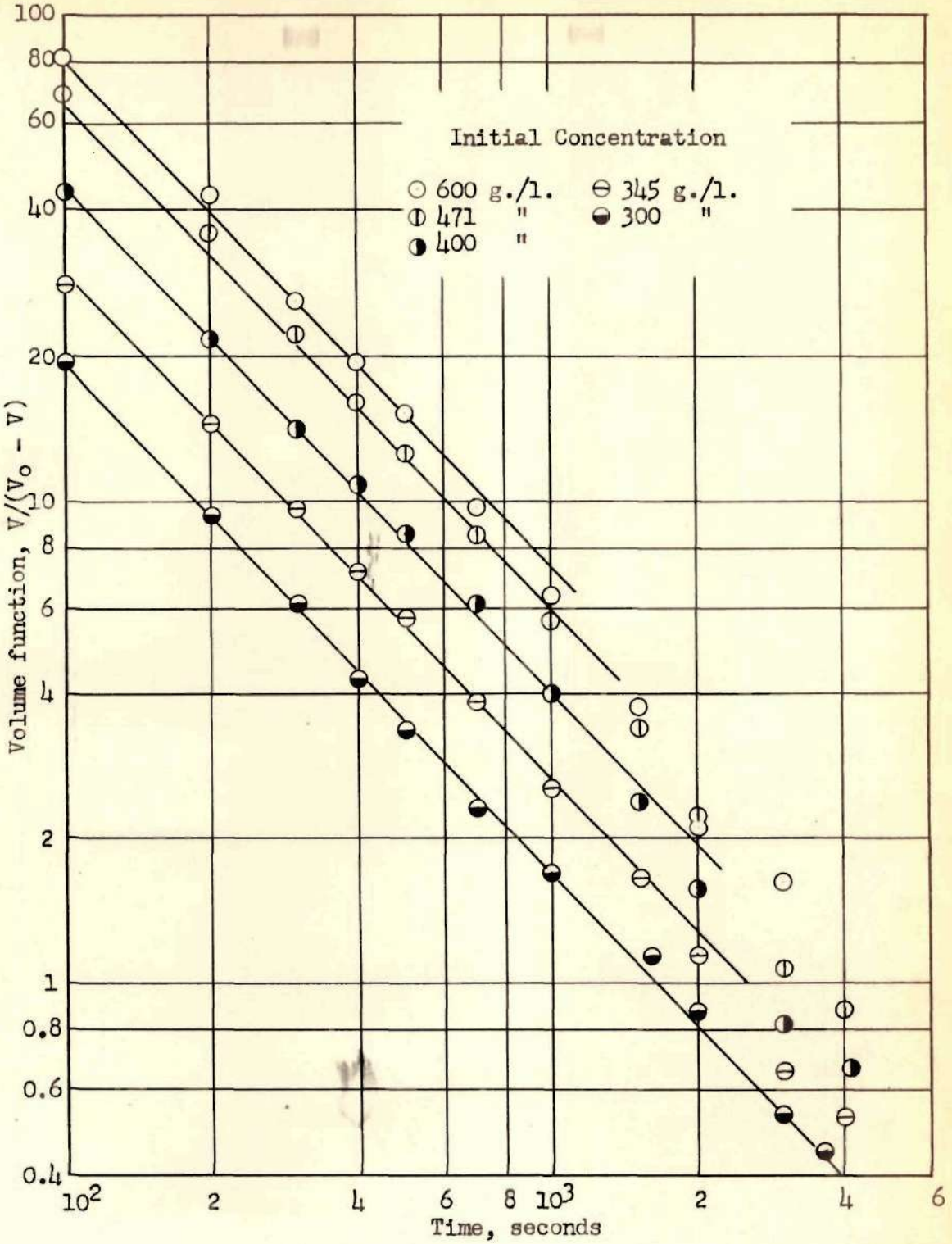


Figure 13. Volume function versus time for silicon carbide - 600

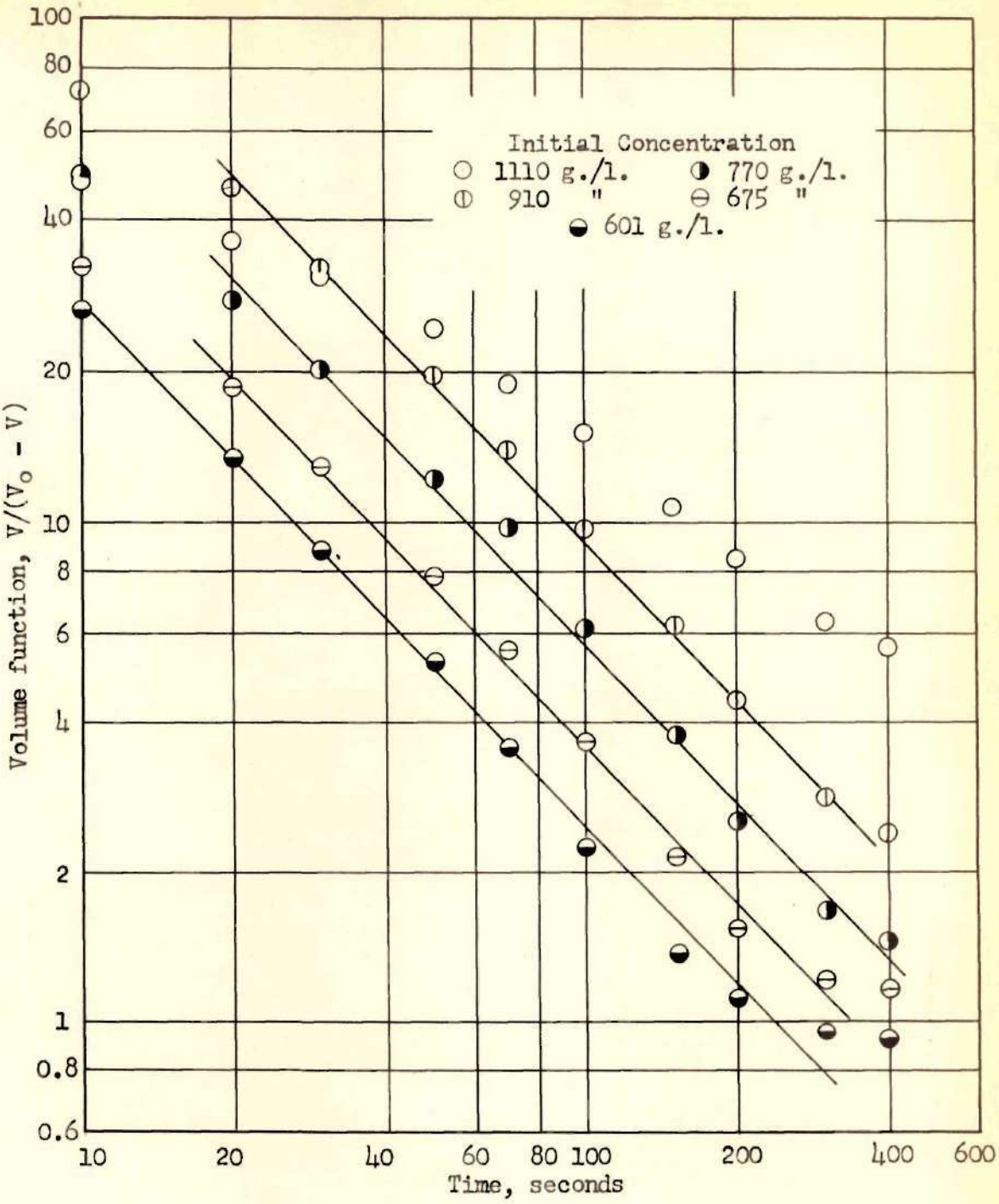


Figure 14. Volume function versus time for potassium perchlorate

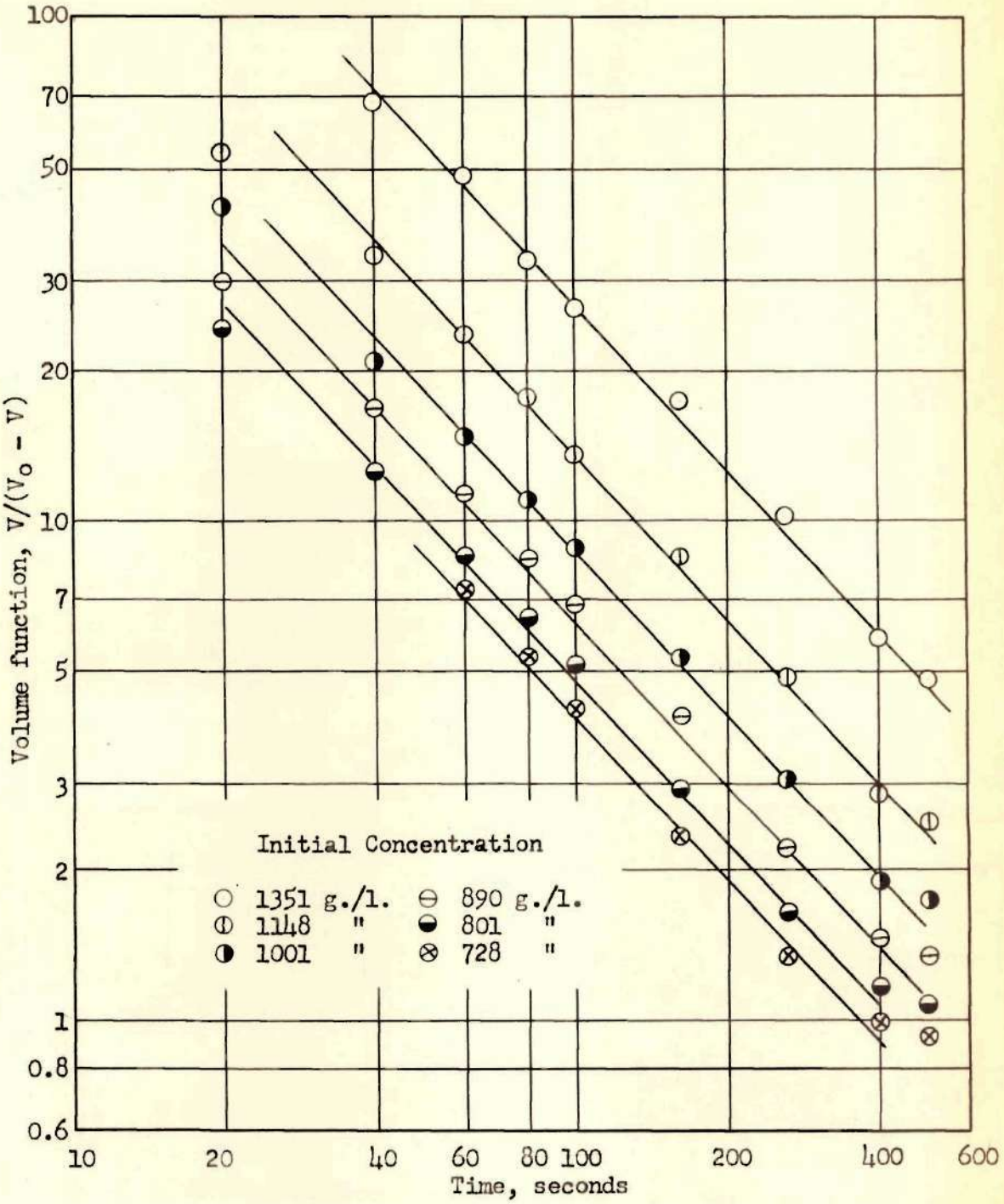


Figure 15. Volume function versus time for potassium sulfate

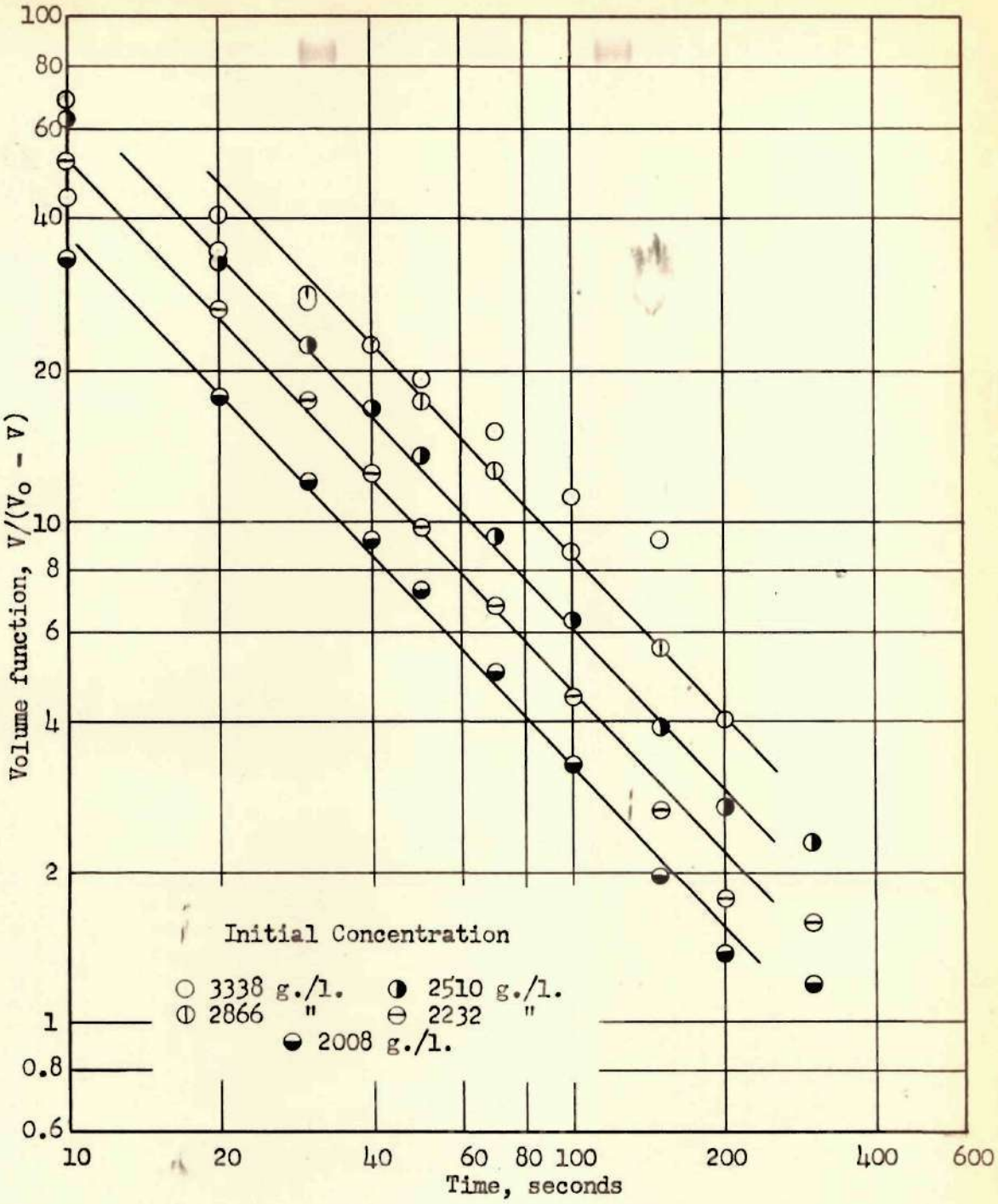


Figure 16. Volume function versus time for mercuric oxide

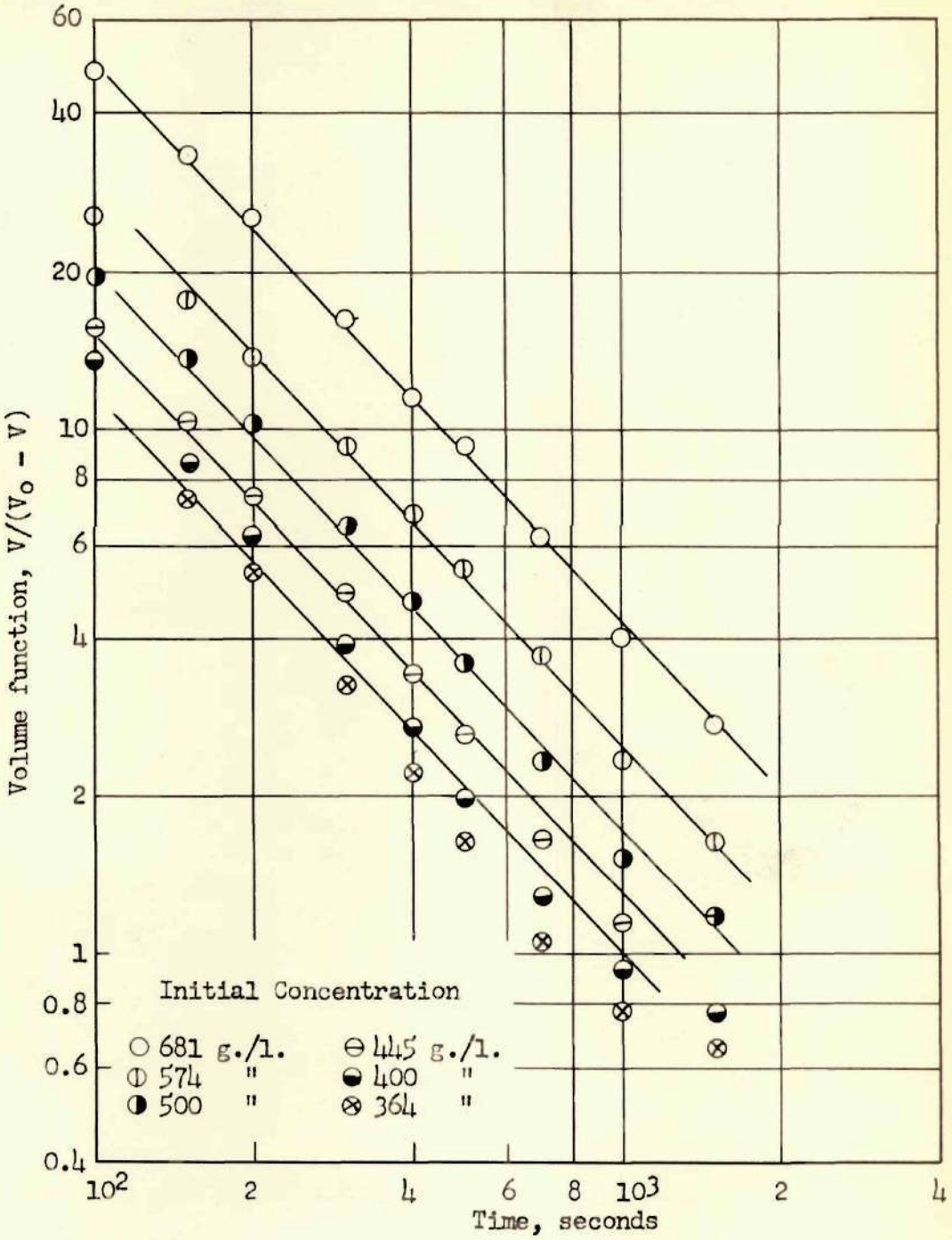


Figure 17. Volume function versus time for manganese dioxide

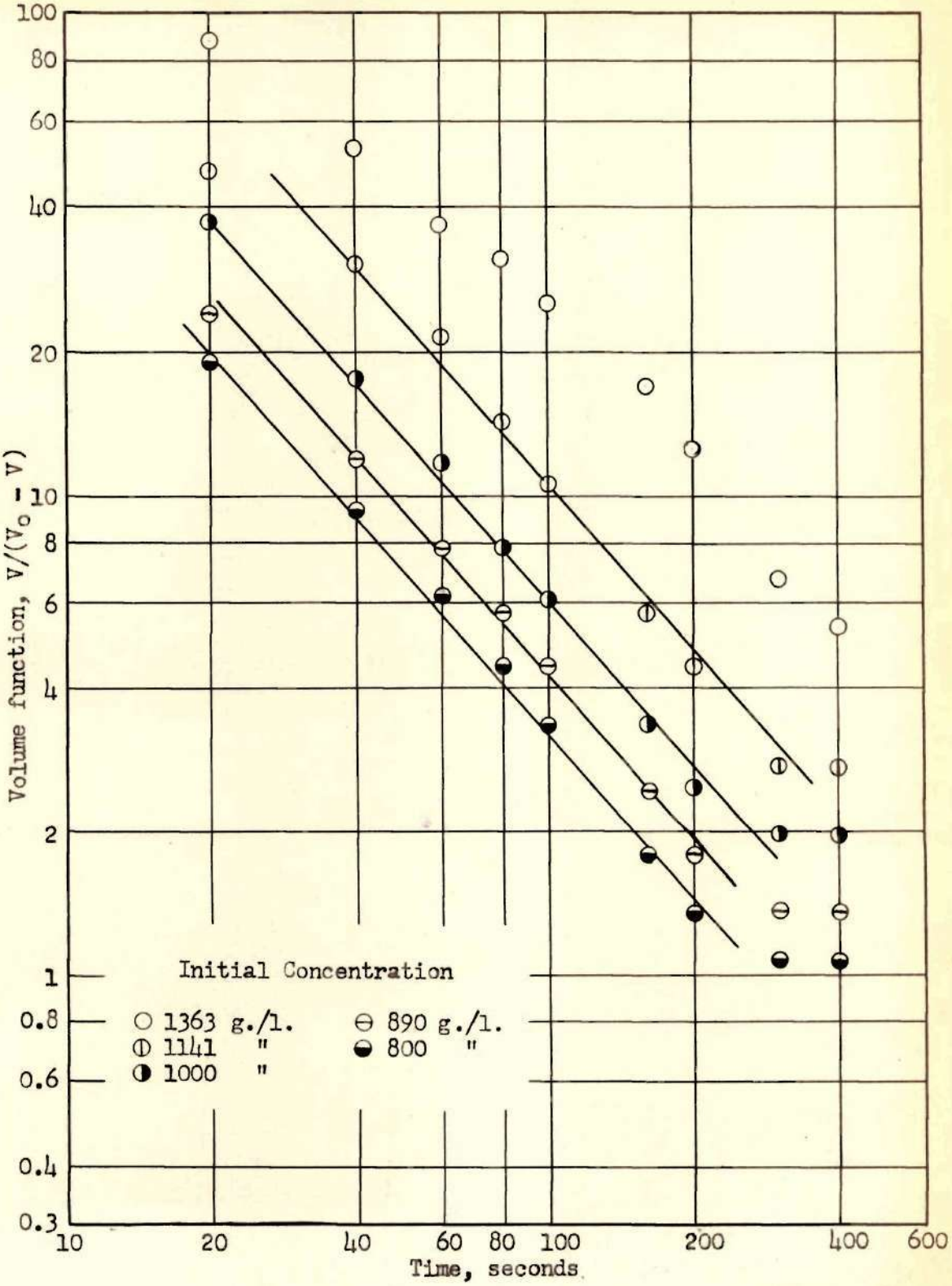


Figure 18. Volume function versus time for powdered iron

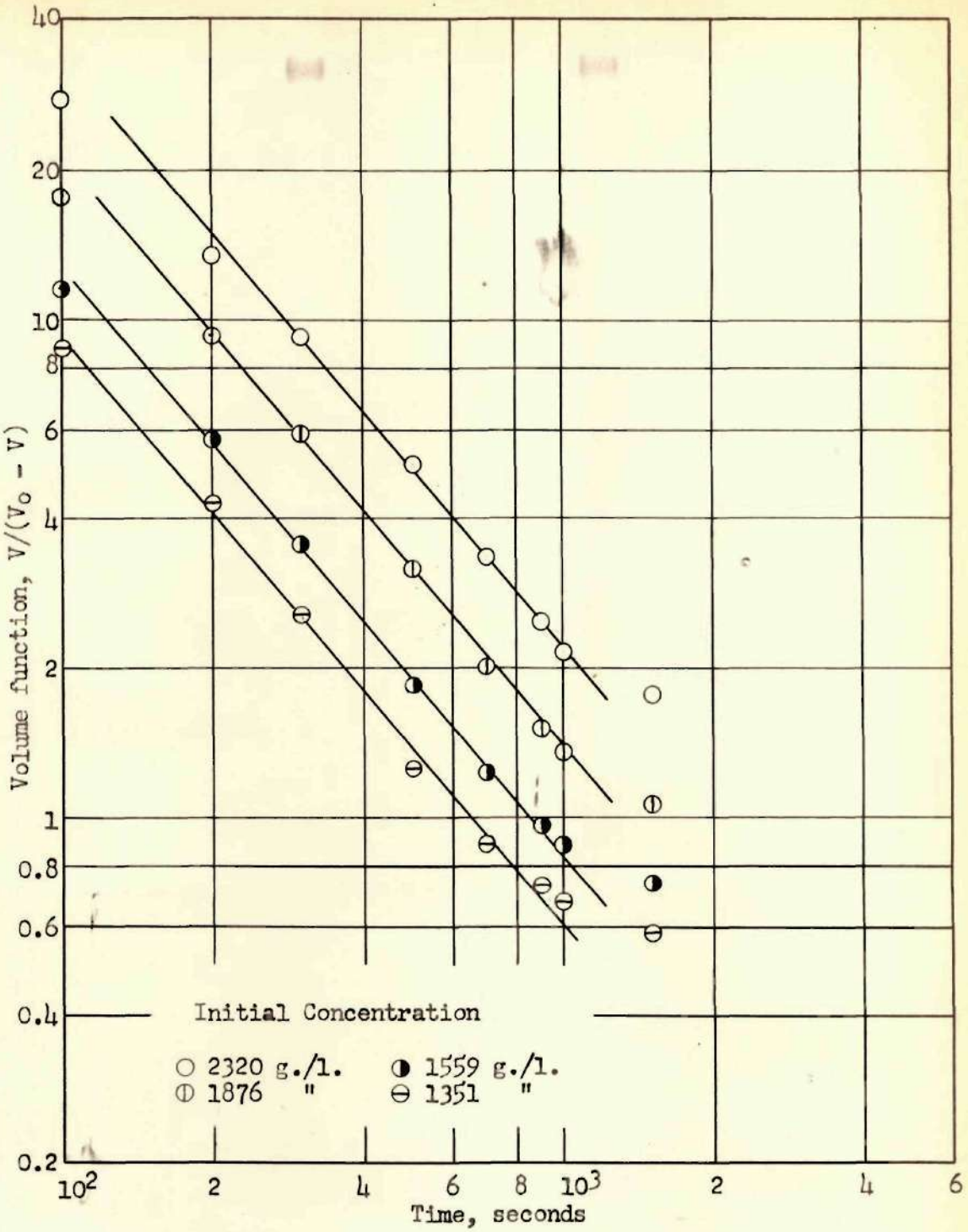


Figure 19. Volume function versus time for powdered zinc

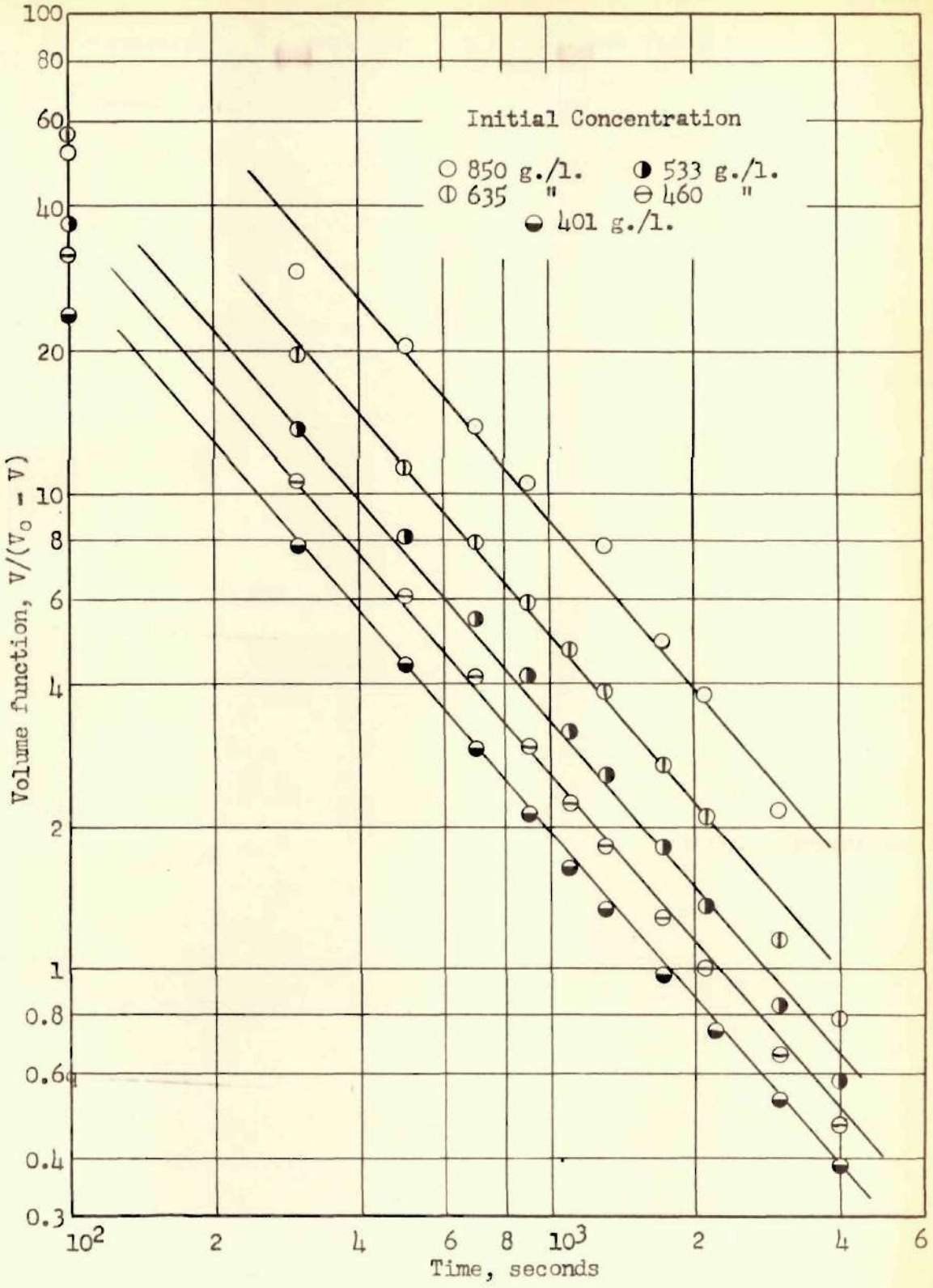


Figure 20. Volume function versus time for levigated alumina

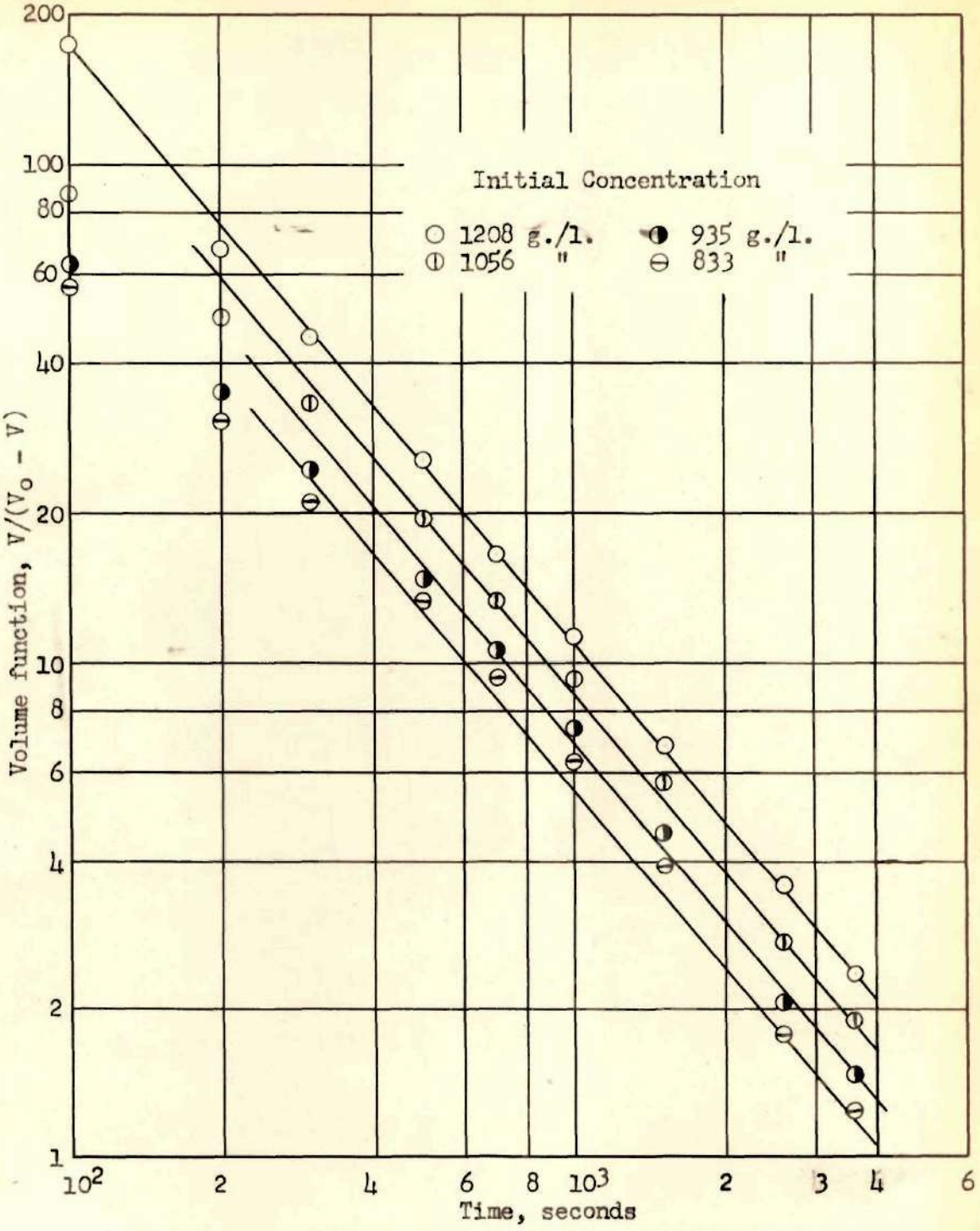


Figure 21. Volume function versus time for calcium flouride

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VITA

Ernest Maxim Peres, Jr., the only son of Bliss Hoyt Peres and Ernest Maxim Peres, Sr., was born on June 17, 1928, in New Orleans, Louisiana. An early evidence of musical talent, whereupon the study of the violin was undertaken, seemed to indicate that a career in the Arts was to be his. However, upon receiving his elementary and secondary education in the public schools of Jefferson Parish, a suburban area of New Orleans, a scholarship to Tulane University inclined him to the study of chemical engineering. His violinistic studies were continued at Loyola University under Dr. Ernest E. Schuyten, throughout his undergraduate work at Tulane. Participation in the numerous musical activities of New Orleans afforded a very enjoyable and profitable source of relaxation during this period.

Completing his engineering studies in June, 1947, after three years of an accelerated program, the degree of Bachelor of Engineering was conferred on him and the 1947 Award of the New Orleans Professional Chapter of the American Institute of Chemical Engineers for the student graduating first in the chemical engineering curriculum was presented to him.

He then went to work in the Asphalt Floor Tile Division of the Flintkote Company in New Orleans. His duties consisted of control, pilot scale development and fundamental research on the addability of the physical properties of high polymeric materials as used in tile. In

1948, he left Flintkote to enter the Massachusetts Institute of Technology for graduate study in chemical engineering. While there, he served as assistant to Dr. E. A. Hauser, the colloid chemist.

After a year at M. I. T., he returned to Tulane University to complete the requirements for the Master of Science degree. The results of his thesis, "An Investigation of the Corrosion Resistance of Anodized and Unanodized Titanium in Acid Media", were published in Industrial and Engineering Chemistry for March of 1951. The Master of Science degree was conferred on him in June of 1950.

He entered the Georgia Institute of Technology in September of 1950 and since then has pursued studies leading to the Doctor of Philosophy degree in chemical engineering. His principle avocations have remained musical (playing viola and violincello in addition to the violin).

He is a member of the American Institute of Chemical Engineers and Alpha Chi Sigma and holds the Socony-Vacuum Fellowship.